

***Sampling and Analysis Plan
for the Post-Decontamination
Characterization of the
WM-103, WM-104, WM-105,
WM-106, and WM-181
Tank Residuals***

March 2004

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March 2004

**Portage Environmental, Inc.
Idaho Falls, Idaho 83403**

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ABSTRACT

The Sampling and Analysis Plan for the Post-Decontamination Characterization of the WM-103, WM-104, WM-105, WM-106, and WM-181 Tank Residuals explains the project description, project organization, and quality assurance and quality control procedures that will be used to sample the residuals remaining in the tank systems following decontamination activities. This document specifies the procedures for obtaining the data of known quality required by the closure activities for the Idaho Nuclear Technology and Engineering Center Tank Farm Facility. The data from this sampling effort will be used to support Idaho Hazardous Waste Management Act/Resource Conservation and Recovery Act closure and the U.S. Department of Energy Tier 1 Closure Plan.

FOREWORD

In 1989 the U.S. Environmental Protection Agency (EPA) published *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA, Interim Final* (EPA 1989). This document stated that a sampling and analysis plan consisted of two separate documents, a field sampling plan (FSP) and a quality assurance project plan (QAPP). In 1998 (revised in 2002), EPA published *EPA Guidance for Quality Assurance Project Plans* (EPA 2002), and in 2001, EPA published *EPA Requirements for Quality Assurance Project Plans* (EPA 2001). These recent documents expand on the guidance provided in the 1989 EPA guidance. Most notably, the 2001 and 2002 documents take the elements defined in the 1989 EPA guidance, which previously required both an FSP and a QAPP to implement, and combine them into one document. Thus, EPA's 2001 and 2002 direction implies that only a single QAPP document is required for each sampling and analysis activity. To alleviate confusion between the old and new nomenclature, this sampling and analysis plan includes all the elements required in a QAPP and FSP, regardless of which EPA guidance is followed. To demonstrate this compliance and to aid readers in locating specific information of interest, two crosswalk tables are provided in Appendix A. The crosswalk tables compare the elements of the EPA 2001 and 2002 guidance, the EPA 1989 requirements, and this document.

CONTENTS

ABSTRACT	iii
FOREWORD	v
1. PROJECT DESCRIPTION	1
1.1 Purpose.....	1
1.2 Background	1
1.3 History of Tanks WM-103 through WM-106 and WM-181.....	2
1.3.1 Tanks WM-103 through WM-106.....	2
1.3.2 Tank WM-181	3
1.3.3 Purpose of Sampling.....	3
2. PROJECT ORGANIZATION AND RESPONSIBILITIES	7
2.1 Project Manager	7
2.2 Environmental Affairs Closure Project Manager.....	8
2.3 Operations Manager	8
2.4 Project Quality Assurance Officer	8
2.5 Job Site Supervisor.....	8
2.6 Field Team Leader	9
2.7 Industrial Hygienist.....	9
2.8 Health and Safety Officer.....	9
2.9 Radiological Engineer.....	10
2.10 Radiological Control Technician	10
2.11 Sampling Team Members	10
2.12 Waste Generator Services – Waste Technical Specialist.....	10
2.13 Data Quality Assessment Chemist/Statistician	11
2.14 Data Storage Administrator.....	11
3. QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA.....	13
3.1 Data Quality Objectives	13

3.1.1	Problem Statement.....	13
3.1.2	Decision Statement	14
3.1.3	Decision Inputs	15
3.1.4	Study Boundaries.....	16
3.1.5	Decision Rule	18
3.1.6	Decision Error Limits	19
3.1.7	Design Optimization.....	22
3.2	Data Quality	28
4.	DOCUMENTATION AND DATA MANAGEMENT.....	31
4.1	Documentation	31
4.1.1	Field Operations Records	31
4.1.2	Laboratory Records	34
4.2	Document Control.....	35
4.3	Data Management	35
5.	SAMPLING PROCESS DESIGN.....	37
5.1	Sample Collection	37
5.1.1	Pre-Sampling Meeting.....	37
5.1.2	Sample Location and Frequency	37
5.1.3	Sample Preservation	42
5.1.4	Field Radiological Control Screening	43
5.1.5	Sample Containers.....	44
5.1.6	Sample Transport.....	44
5.1.7	Waste Management	45
6.	SAMPLING PROCEDURES.....	47
6.1	Simple Sampler	47
6.2	Submersible Pump	47
6.3	Sample Collection Procedures	48
7.	ANALYTICAL METHODS	51
7.1	Analytical Laboratory	51
8.	INSTRUMENT CALIBRATION PROCEDURES	63
8.1	Laboratory Instrument Calibration.....	63

8.2	Field Equipment Calibration/Setup.....	63
8.3	Preventative Maintenance Procedures and Frequency.....	63
9.	DATA VALIDATION AND REPORTING	65
9.1	Data Reduction.....	65
9.2	Data Validation	65
9.3	Data Quality Assessment	66
9.4	Data Use.....	66
9.5	Reporting.....	66
10.	INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY	67
10.1	Laboratory Quality Control.....	67
10.2	Field Quality Control	67
10.3	Inspection/Acceptance Requirements for Supplies and Consumables.....	67
11.	SYSTEM AND PERFORMANCE ASSESSMENTS, FREQUENCY AND CORRECTIVE ACTIONs	69
11.1	System and Performance Assessments	69
11.2	Corrective Action	69
	11.2.1 Laboratory Corrective Action.....	69
	11.2.2 Field Corrective Action	70
11.3	Reports to Management	70
12.	REFERENCES	71
Appendix A—Crosswalk between the Environmental Protection Agency QAPP and FSP Requirements and the SAP for Post-Decontamination Characterization of WM-103 through WM-106 and WM-181 Tank Residuals		75

FIGURES

1.	Example of a decision performance goal diagram and corresponding decision error limits table	22
2.	Riser locations where samples will be collected from WM-103, WM-104, WM-105, and WM-106.....	38
3.	Riser locations where samples will be collected from WM-181	38

TABLES

1.	Results of 1990 sampling of the 30,000-gal tanks.....	5
2.	Key project responsibilities and responsible personnel.....	7
3.	Required sample size (n) associated with various false-positive error rates (α) and false-negative error rates (β) when $\sigma = 12.5\%$ of the action level and the width of the gray region is from 80% to 100% of the action level (i.e., $\Delta = 0.20$).....	27
4.	Summary of analysis requirements for solid residuals remaining in Tank WM-103 through WM-106 and WM-181 system components following decontamination	29
5.	Summary of analysis requirements for liquid residuals remaining in Tank WM-103 through WM-106 and WM-181 system components following decontamination	30
6.	Summary of sample collection, holding time, and preservation requirements for radiological analyses	40
7.	Summary of sample collection, holding time, and preservation requirements for nonradiological analyses	41
8.	Anticipated sample collection from each of the WM-103 through WM-106 and WM-181 tank systems.....	50
9.	Analytical method source documents and method descriptions.....	52
10.	Sample preparation, analytical methods, and recommended detection limits—solids.....	53
11.	Sample preparation, analytical methods, and recommended detection limits—liquids	57

ACRONYMS

ACMM	Analytical Chemistry Methods Manual
ALARA	as low as reasonably achievable
ARDC	administrative record and document control
CFR	Code of Federal Regulations
COC	chain of custody
CVAA	cold vapor atomic adsorption
DOE	U.S. Department of Energy
DQA	data quality assessment
DQO	data quality objective
DVB	diversion valve box
EPA	U.S. Environmental Protection Agency
EQL	estimated quantitation limit
ESH&Q	environmental, safety, health, and quality
FSP	field sampling plan
FTL	field team leader
GC	gas chromatography
GFAA	graphite furnace atomic adsorption
HDPE	high-density polyethylene
HSO	health and safety officer
HWMA	Idaho Hazardous Waste Management Act
IDEQ	Idaho Department of Environmental Quality
IH	industrial hygienist
INEEL	Idaho National Engineering and Environmental Laboratory

INTEC	Idaho Nuclear Technology and Engineering Center
ISD	interim status document
JSS	job site supervisor
LLW	low-level waste
MCP	management control procedure
MS	mass spectrometry
N/A	not applicable
ND	not detected
PA	performance assessment
PCB	polychlorinated biphenyl
PE	professional engineer
PEWE	process equipment waste evaporator
PM	project manager
PQAO	project quality assurance officer
PRD	program requirements document
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RAL	Remote Analytical Laboratory
RCRA	Resource Conservation and Recovery Act
RCT	radiological control technician
SAP	sampling and analysis plan
SOW	statement of work
SVOA	semivolatile organic analysis

SVOC	semivolatile organic compound
TBD	to be determined
TCLP	toxicity characteristic leaching procedure
TFF	Tank Farm Facility
UCL	upper confidence limit
USC	United States Code
VCO	Voluntary Consent Order
VOA	volatile organic analysis
VOC	volatile organic compound

Sampling and Analysis Plan for the Post-Decontamination Characterization of the WM-103, WM-104, WM-105, WM-106, and WM-181 Tank Residuals

1. PROJECT DESCRIPTION

1.1 Purpose

This sampling and analysis plan (SAP) describes the sampling, analysis, and quality assurance (QA) and quality control (QC) procedures to be used for the characterization of the post-decontamination residuals remaining in the tanks, WM-181 vault sump, and cooling coils for Tanks WM-103, WM-104, WM-105, WM-106, and WM-181. These tanks are a part of the Idaho Nuclear Technology and Engineering Center (INTEC) Tank Farm Facility (TFF) at the Idaho National Engineering and Environmental Laboratory (INEEL).

This SAP is a combined quality assurance project plan (QAPP) and field sampling plan (FSP) in accordance with U.S. Environmental Protection Agency (EPA) guidance (EPA 2001, 2002). The elements of a QAPP present the activities, organization, and QA/QC protocols to achieve the data quality objectives (DQOs) of the sampling and analysis effort. The elements of an FSP specify sampling and analyses required to ensure compliance with the regulatory requirements for closure as defined by the Idaho Hazardous Waste Management Act (HWMA) (State of Idaho 1983) and the Resource Conservation and Recovery Act (RCRA) (42 United States Code [USC] 6901 et seq., 1976) and with U.S. Department of Energy (DOE) closure requirements (DOE G 435.1-1, 1999; DOE M 435.1-1, 2001; DOE O 435.1, 2001). This SAP is based on the requirements stated in the EPA *Guidance for Quality Assurance Project Plans*^a (EPA 2002). This SAP also will ensure compliance with the QA/QC requirements of DOE's management and operations contractor, Bechtel BWXT Idaho, LLC; EPA Region 10; DOE Idaho Operations Office; DOE Headquarters; and Idaho Department of Environmental Quality (IDEQ). This plan will serve as the governing document for all activities conducted in support of the post-decontamination characterization of the residuals present in WM-103 through WM-106 and WM-181 tank system components.

1.2 Background

The TFF includes 11 belowground 300,000-gal and 318,000-gal tanks (hereinafter referred to as 300,000-gal tanks) and four 30,000-gal tanks. Each 300,000-gal tank, numbered WM-180 through WM-190, is enclosed within a concrete vault. The four 30,000-gal tanks, numbered WM-103 through WM-106, sit on concrete pads. The TFF was designed primarily to receive liquid wastes from nuclear fuel reprocessing operations at the Idaho Chemical Processing Plant, now called INTEC. Reprocessing operations to recover ²³⁵U began in 1953 and ceased in 1992. The liquid wastes were stored in the tanks for eventual solidification into a granular calcine at the Waste Calcining Facility and later at the New Waste Calcining Facility. The TFF currently receives liquids from the process equipment waste

a. To demonstrate compliance with EPA requirements and guidance documents, as stated in the Foreword, and to aid readers in locating specific information of interest, two crosswalk tables are provided in Appendix A. These tables compare the elements of the EPA 2001 and 2002 guidance, EPA 1989 requirements, and this document.

evaporator (PEWE). The liquids are derived from waste produced by plant operations such as fuel storage, sample analysis, off-gas cleanup, and equipment and facility decontamination.

Because the tanks at the TFF do not meet HWMA/RCRA secondary containment requirements and a need for such storage is not evident after 2012, the TFF is being closed in phases beginning in 2003. The first phase of the closure included Tanks WM-182 and WM-183, which served as a proof-of-process demonstration of waste removal, decontamination, and sampling techniques. Data pertaining to the closure of Tanks WM-182 and WM-183 have been collected and indicate successful tank decontamination to levels compliant with HWMA/RCRA and DOE requirements for TFF site closure. (See *Idaho Hazardous Waste Management Act/Resource Conservation and Recovery Act Closure Plan for Idaho Nuclear Technology and Engineering Center Tanks WM-184, WM-185, and WM-186*, Appendix B, for post-decontamination data [DOE-ID 2003a].) The second phase of closure included Tanks WM-184, WM-185, and WM-186, as well as associated ancillary system features. Phase III closure includes the four 30,000-gal tanks (Tanks WM-103 through WM-106) and WM-181, a 318,000-gal tank.

1.3 History of Tanks WM-103 through WM-106 and WM-181

1.3.1 Tanks WM-103 through WM-106

Tanks WM-103 through WM-106 are 30,750-gal (referred to as the 30,000-gal tanks) stainless steel tanks built from 1953 through 1955. The tanks were constructed on reinforced concrete pads and directly buried approximately 15 ft below grade. The pad for each tank has a 1-ft-high by 9-in.-wide concrete perimeter curb equipped with a 60-gal sump. Any liquid collected on the pad drains to the sump. Tanks WM-103 through WM-106 contain stainless steel, closed-loop cooling coils to control the temperature of liquid wastes and minimize tank corrosion. Tank WM-106 contains 12 cooling coils and the remaining tanks each contain four; however, the cooling coils for these tanks are all part of the same, continuous system and will, therefore, be sampled as a single system. Four risers in each tank extend to ground level to provide access. Each tank is equipped with two to four steam jets.

Tanks WM-103 through WM-106 were designed to hold the first-cycle waste from fuel reprocessing activities. Both stainless steel and zirconium-type fuel claddings were processed as part of these activities. Sulfuric acid and hydrofluoric acid were used in the dissolution of stainless steel and zirconium-type fuel claddings, respectively. In September/October 1982, condensate from the PEWE system was transferred into Tanks WM-103 through WM-105 using temporary aboveground piping because the service waste system injection well was out of service. The temporary piping was removed after the injection well was refurbished. The tanks were emptied to their heels and taken out of service in 1983.

Tank WM-106 was used to collect stainless steel-clad fuel reprocessing waste until January 1974, at which time it was emptied. The tank was flushed in January 1975 and November 1982. Tank WM-106 was emptied of its contents and the tank flushed before implementation of RCRA. Tank WM-106 has not received any RCRA waste since the implementation of RCRA and has been determined to be nonhazardous per RCRA regulations (EDF-2614, 2002). Nonhazardous steam condensate from a leaky valve was received into the tank from 1990 until 2001. Though WM-106 does not fall within the closure requirements of RCRA, it will be sampled and grouted in the same fashion as the other three 30,000-gal tanks.

In June 2000, IDEQ and DOE entered into a Consent Order (IDEQ 2000) regarding the INEEL Site. The Consent Order, or Voluntary Consent Order (VCO), is a long-term agreement between the State of Idaho and DOE to resolve potential compliance issues with provisions of the HWMA (State of Idaho 1983)/RCRA (42 USC 6901 et seq., 1976) at the INEEL Site.

The VCO Action Plan documents the actions to be taken and the milestones for covered matters under the VCO (IDEQ 2000). The Action Plan is further separated into detailed action plans that address specific compliance issues. Tanks WM-103, WM-104, WM-105, and WM-106 are included in the SITE-TANK-005 Action Plan of the VCO. The SITE-TANK-005 Action Plan addresses tanks/components that require a hazardous waste determination or need to be verified as empty.

Tanks WM-103, WM-104, and WM-105 were characterized as having managed HWMA/RCRA-hazardous waste; Tank WM-106 was characterized as not having managed HWMA/RCRA-hazardous waste (EDF-2614, 2002). In accordance with the SITE-TANK-005 Action Plan, interim actions and a further milestone were identified for the tanks. In September 2002, the tanks were emptied as an interim action under the VCO to the maximum extent possible to reduce the potential for an inadvertent release since the tanks do not have secondary containment meeting the secondary containment requirements of HWMA/RCRA. An enforceable milestone was established under the SITE-TANK-005 Action Plan for the submittal of a HWMA/RCRA closure plan addressing tanks WM-103, WM-104, and WM-105 to the State of Idaho by September 30, 2007.

In 1990, raw water was added to Tanks WM-103 through WM-106 to provide enough solution to sample. After samples were collected, the tanks were emptied to their heels and the contents used to flush lines from the TFF to the PEWE system. At this time, the inlet lines were cut and capped. However, the outlet lines remained open and operational.

1.3.2 Tank WM-181

Tank WM-181 is one of two 318,000-gal stainless steel tanks designed and built between 1951 and 1952. This tank is contained in a monolithic octagonal vault, which contains one sump area to allow liquids to drain. The sump is emptied using a steam jet accessed through a vault or sump riser. Five 12-in. risers extend to ground level to provide access to the tank. Unlike Tanks WM-103 through WM-106, WM-181 has no cooling capabilities.

Tank WM-181 was primarily used to store sodium-bearing waste, which includes the liquid remaining from the second and third extraction cycles and solution resulting from decontamination activities after first being concentrated by evaporation in the PEWE. This waste is generally referred to as sodium-bearing waste because it is high in sodium content from decontamination activities.

1.3.3 Purpose of Sampling

The overall purpose of the post-decontamination sampling and analysis for the WM-103 through WM-106 and WM-181 tank system residuals is to

- Show that hazardous wastes are not left in place in the TFF tank system. With the exception of WM-106, wastes previously stored in the tanks were hazardous waste (as determined by the toxicity and corrosivity characteristic). Therefore, the mean characteristic of the post-decontamination residues remaining in each individual tank must be shown to be less than the toxicity characteristic (40 Code of Federal Regulations [CFR] 261.24, Table 1, 2004) and have a pH between 2 and 12.5.
- Determine whether or not the post-decontamination mean concentrations of hazardous constituents remaining in the TFF meet the HWMA/RCRA clean-closure action levels specified in the *Idaho Hazardous Waste Management Act/Resource Conservation and Recovery Act Closure Plan for Idaho Nuclear Technology and Engineering Center Tanks WM-103, WM-104, WM-105, WM-106, and WM-181* (DOE-ID 2004), hereinafter referred to as the HWMA/RCRA closure plan.

- Determine whether the residuals remaining in the TFF tank systems have activities that meet DOE Order 435.1 (2001) radioactive waste management performance assessment (PA) requirements for closure of the facility.

Samples from the post-decontamination residuals in the WM-103 through WM-106 and WM-181 tanks and residuals of the final rinse solutions present in the WM-181 vault sump must be collected and analyzed for a group of parameters. The samples must pass specific criteria to satisfy HWMA/RCRA and DOE requirements for TFF site closure. No waste transfer lines will be sampled for Tanks WM-103 through WM-106 and WM-181. Rinsates collected from associated waste transfer lines were described in the *Sampling and Analysis Plan for the Post-Decontamination Characterization of the Process Waste Lines from INTEC Tank Farm Facility Tanks WM-182 and WM-183* (INEEL 2001) and are assumed to represent all waste transfer lines in the TFF.

Because cooling coils are present in Tanks WM-103 through WM-106, additional samples of post-decontamination residuals will be taken from the cooling coils and analyzed for chromium, pH, and gamma. It is not known if the cooling coils contained chromium as a corrosion inhibitor. It is known that the contents of the cooling coils never came in contact with the tank waste. Therefore, chromium is the only analyte of interest in the cooling coil rinsates. A specific action level for the chromium in these rinsates is established, and only chromium data from the analyses of the cooling coil rinsates will be used in assessing whether or not TFF residuals meet the HWMA/RCRA clean closure action levels.

Sampling efforts conducted in 1999 and 2000 yielded data about WM-182 and WM-183 tank wastes before the start of decontamination activities. These initial waste characterization data are analyzed and summarized in Section 3.2 of the *Sampling and Analysis Plan for the Post-Decontamination Characterization of the WM-182 and WM-183 Tank Residuals* (INEEL 2002). No comprehensive pre-decontamination characterization data exist for Tanks WM-103 through WM-106 and WM-181. However, in 1990, the 30,000-gal tank heels were sampled and analyzed for radionuclides and RCRA-hazardous materials. The analytical results indicated contaminant concentrations of organics that were below RCRA limits for hazardous waste and a small amount of radioactivity.^b To date, this is the only information available concerning potential hazardous and radioactive contamination in the 30,000-gal tanks. Table 1 lists analytical results for metals from the 1990 water samples collected from the 30,000-gal tanks.

b. Matule, A. J., WINCO, Personal Communication to D. C. Machovec, WINCO, "Solid Sampling of WM 103–106," AJM-20-90, September 26, 1990.

Table 1. Results of 1990 sampling of the 30,000-gal tanks.

Analyte	Tank WM-103 Concentration (mg/L)	Tank WM-104 Concentration (mg/L)	Tank WM-105 Concentration (mg/L)	Tank WM-106 Concentration (mg/L)
pH (unitless)	3.4	3.4	6.0	7.9
Arsenic	ND ^a	ND	ND	ND
Barium	0.46	0.11	0.11	0.27
Cadmium	ND	ND	ND	ND
Chromium	0.24	0.84	0.04	0.05
Lead	ND	ND	ND	ND
Mercury	0.005	0.004	0.007	0.003
Selenium	ND	ND	ND	ND
Silver	ND	0.005	0.006	ND
Cs-137	1.08E+04 pCi/mL		5.19E+03 pCi/mL	

a. ND = Not detected.

2. PROJECT ORGANIZATION AND RESPONSIBILITIES

The closure of tank systems WM-103 through WM-106 and WM-181 has a clearly defined project organization. This will ensure that project closure objectives, data gathering and reporting, data evaluation and interpretation, closure design, and operational safety meet INEEL requirements. Table 2 lists project personnel and their responsibilities. The table is not intended to imply that a separate individual is required for each project role listed. One individual may perform more than one project role. The following subsections outline the specific duties of the project personnel associated with each role throughout the post-decontamination characterization effort.

Table 2. Key project responsibilities and responsible personnel.

Project Role	Responsible Official	Telephone Number
Project Manager	Keith Quigley	526-3779
Environmental Affairs Closure Project Manager	Susan Evans	526-0186
Operations Manager	Frank Ward	526-3010
Project Quality Assurance Officer	TBD ^a	
Job Site Supervisor	TBD	
Field Team Leader	TBD	
Industrial Hygienist	TBD	
Health and Safety Officer	TBD	
Radiological Engineer	TBD	
Radiological Control Technician	TBD	
Sampling Team Member ^b	TBD	
Waste Generator Services – Waste Technical Specialist	TBD	
Data Quality Assessment Chemist/Statistician	TBD	
Data Quality Assessment Chemist/Statistician	TBD	
Data Storage Administrator	TFF Closure Project	

a. TBD = To be determined.

b. All sampling team members will be identified before sampling begins.

2.1 Project Manager

The project manager (PM) ensures that all activities conducted during the project comply with INEEL management control procedures (MCPs) and program requirements documents (PRDs) and all applicable requirements of the Occupational Safety and Health Administration, EPA, DOE, U.S. Department of Transportation, and State of Idaho. The PM coordinates all document preparation, field and laboratory activities, data evaluation, risk assessment, dose assessment, and closure design activities. The PM is responsible for the overall work scope, schedule, and budget.

The PM is responsible for field activities and for all personnel (including craft personnel) assigned to work at the project location. The PM serves as the interface between operations and project personnel and works closely with the sampling team at the site to ensure that the objectives of the project are

accomplished in a safe and efficient manner. The PM works with all other identified project personnel to accomplish day-to-day operations at the site, identify and obtain additional resources needed at the site, and interact with the INTEC environment, safety, health, and quality (ESH&Q) oversight personnel on matters regarding health and safety.

2.2 Environmental Affairs Closure Project Manager

The Environmental Affairs closure PM is responsible for regulatory oversight of the project. The Environmental Affairs closure PM ensures that closure documentation complies with regulatory requirements and acts as the main resource for project communication to the independent professional engineer (PE) who certifies closure. Any deviation from the requirements specified in closure plan documentation will be communicated to the PE through the Environmental Affairs closure PM.

2.3 Operations Manager

The TFF operations manager is responsible for all work that is accomplished in the facility. This includes ensuring that work activities are scheduled, adequate safety and health support personnel are available, and that the work performed is completed by personnel that are adequately trained to accomplish the work. The operations manager is a key function of the Integrated Safety Management System at the INEEL.

2.4 Project Quality Assurance Officer

The project quality assurance officer (PQAO) reports directly to INEEL management and is organizationally independent for all post-decontamination tank system characterization and closure activities for Tanks WM-103 through WM-106 and WM-181. The PQAO is also responsible for the control and implementation of all QA/QC actions conducted during post-decontamination characterization and subsequent closure activities. These actions include:

- Conducting QA oversight of all reporting and all project data-gathering efforts
- Conducting QA oversight for all laboratory analysis and data reporting
- Conducting QA oversight for all data validation and data evaluation
- Identifying and reporting any deviations from project QA objectives
- Identifying any necessary corrective actions
- Monitoring the performance of all field sampling activities (sample collection, decontamination, and transport)
- Conducting system and performance audits, if necessary
- Preparing and submitting QA reports to management.

2.5 Job Site Supervisor

The job site supervisor (JSS) serves as the representative for the TFF closure project at the site. The JSS manages field activities, craft personnel, and other personnel assigned to work at the site. The JSS is

the interface between operations and project personnel and works closely with the sampling team at the site to ensure that the objectives of the project are accomplished in a safe and efficient manner. The JSS and the PM work together to accomplish day-to-day operations at the site, identify and obtain additional resources needed at the site, and interact with the health and safety officer (HSO), industrial hygienist (IH), and radiological control technician (RCT) on matters regarding health and safety. The JSS will be informed about any health and safety issues that arise at the site and may stop work at the site if an unsafe condition exists. The JSS participates in all daily pre-job briefings. The duties of the JSS may be combined with the duties of the field team leader (FTL) and be performed by one individual.

2.6 Field Team Leader

The FTL is the INEEL representative at the site with responsibility for the safe and successful completion of sampling the post-decontamination tank heels from Tanks WM-103 through WM-106 and WM-181. The FTL works with the JSS, RCT, and field team to manage field sampling operations and execute the SAP. The FTL enforces site control, documents activities, and is responsible for ensuring the daily safety briefings at the start of the shift occur as required. The FTL may personally conduct the daily safety briefings if necessary. Health and safety issues may be brought to the attention of the FTL. As previously stated, the duties of the FTL may be combined with the duties of the JSS and be performed by one individual.

If the FTL leaves the site, an alternate will be appointed to act as the FTL. The identity of the acting FTL will be conveyed to site personnel, recorded in the sampling logbook, and communicated to the facility representative, when appropriate.

2.7 Industrial Hygienist

The IH is the primary source for information regarding hazardous and toxic agents at the project site. The IH assesses the potential for worker exposures to hazardous agents according to applicable procedures, MCPs, and accepted industry industrial hygiene practices and protocol. By participating in sampling, the IH assesses and recommends appropriate hazard controls for the protection of project personnel and operates and maintains personnel sampling and monitoring equipment. The IH also recommends and assesses the use of personnel protective equipment in the health and safety plan or other health and safety documentation such as safe work permits or radiological work permits.

In the event of a general area evacuation, the IH, in conjunction with other recovery team members, will assist the JSS and PM in determining whether conditions exist for safe site reentry. Personnel who have been exposed to hazardous agents or show signs and symptoms of health effects resulting from possible exposure to hazardous agents will be referred to an Occupational Medical Program physician by the IH, the individual's supervisor, or the HSO. The IH may have other duties at the site as specified in other procedures. During emergencies involving hazardous materials, the IH will be responsible for coordinating airborne sampling and monitoring results with members of the Emergency Response Organization.

2.8 Health and Safety Officer

The HSO serves as the primary contact for health and safety issues. A specific individual designated as the HSO may not be necessary because of the current health, safety, and radiological controls staff at INTEC. The PM will determine if an HSO is needed for this project. The HSO advises the JSS and FTL on all aspects of health and safety. The HSO is authorized to stop work at the project site if any operation threatens worker or public health or safety. The HSO is authorized to verify compliance

with health and safety procedures, conduct inspections, require and monitor corrective actions, and monitor decontamination procedures and require corrections, as appropriate. The HSO is supported by ESH&Q professionals at the INEEL (safety engineers, IHs, RCTs, radiological engineers, environmental coordinators, and facility representatives), as necessary.

A person assigned as the HSO (or as an acting HSO) must be qualified to recognize and evaluate hazards (in accordance with 29 CFR 1910.120(a)(3), 2003) and will have the authority to take or direct actions to ensure that workers are protected. If the HSO must leave the site, an alternate, the IH, or the FTL will be appointed by the HSO as acting HSO. The identity of the acting HSO will be recorded in the appropriate logbooks and site personnel will be notified.

2.9 Radiological Engineer

The radiological engineer is the primary source of radiological information and provides guidance on radiological hazards and mitigations. The radiological engineer's responsibilities include evaluating the intended work, conducting as low as reasonably achievable (ALARA) reviews, giving input to work packages and radiological work permits, and establishing radiological hold points.

2.10 Radiological Control Technician

The RCT is the primary source for information and guidance on radiological hazards and is present at the project site during all operations. Responsibilities of the RCT include radiologically surveying the project site, equipment, and samples; providing guidance for radioactive decontamination of equipment and personnel; and, if significant radiological contamination occurs, accompanying any affected personnel to the nearest INEEL medical facility for evaluation. The RCT notifies the JSS of any radiological occurrence that must be reported as directed by PRD-183, "INEEL Radiological Control Manual" (2000). The RCT may have other duties at the site as specified in other procedures.

2.11 Sampling Team Members

The sampling team will be fully trained and skilled in the operation of the simple sampler, submersible pump, or other appropriate sampling equipment. The sampling team members will be responsible for operating the sampling equipment, including collecting samples in sufficient numbers and volumes to meet the requirements presented in this SAP. The sampling team will ensure that the sampling equipment is ready for the sampling event according to the appropriate standard operating procedure(s).

Sampling team members must be experienced in all aspects of sampling the TFF tanks as well as in the requirements of INTEC and INEEL ESH&Q procedures and policies. Sampling personnel must also be familiar with the TFF systems and components.

2.12 Waste Generator Services – Waste Technical Specialist

The INEEL Waste Generator Services waste technical specialist will ensure the disposal of non-sample waste material complies with Section 6 of the approved HWMA/RCRA closure plan (DOE-ID 2004), and that applicable paperwork is completed. All samples and analysis wastes disposed of by the INEEL Analytical Chemistry Laboratory will be disposed of to the PEWE system through normal routes or in accordance with INEEL MCP-3480, "Environmental Instructions for Facilities, Processes, Materials and Equipment" (2002). The Waste Generator Services waste technical specialist will ensure compliance with the applicable HWMA/RCRA requirements, Interim Status Document (ISD)-9, "INTEC RCRA Interim Status Document for the Process Equipment Waste Evaporator System, Section B – Waste

Analysis Plan” (2002), MCP-62, “Waste Generator Services Low-Level Waste Management” (2003), and MCP-70, “Mixed Low-Level Waste Management” (2003).

2.13 Data Quality Assessment Chemist/Statistician

The data quality assessment (DQA) process is performed by one (or more) chemist/statistician familiar with analytical chemistry, statistical sampling designs, and statistical hypothesis testing. Steps of the DQA process involve data plotting, testing for outlying data points, and statistical hypothesis testing relative to the null and alternative hypotheses stated in the DQOs. The outcome of the DQA process is a statement that the statistical hypothesis testing suggests that the null hypothesis is accurate, that the null hypothesis has been rejected, or that not enough data exist to make a determinative conclusion based upon the hypothesis test used. In the latter case, either additional data must be collected to support the statistical hypothesis testing or the data user must make a decision with higher uncertainty than the levels expressed in the DQOs.

Data that are not necessarily invalid may be flagged during the data validation process. Flagged data are reviewed during the DQA process to determine whether the validation flags affect the intended use of the data. The DQA chemist/statistician will document whether or not flagged data are used in statistical hypothesis testing in the DQA report.

2.14 Data Storage Administrator

The data storage administrator is responsible for maintaining the project administrative record and document control (ARDC). The ARDC will be the official repository for all TFF closure project records. Upon completion of the WM-103 through WM-106 and WM-181 post-decontamination tank system characterization, the PM will transfer all hard-copy information and documentation developed from the project to the designated ARDC for appropriate archiving. Hard-copy information and documentation include field logbooks, field and laboratory chain-of-custody (COC) forms, laboratory reports and data, engineering calculations and drawings, final design reports, data validation reports, DQA reports, and all other technical reports related to the project. Copies of all analytical data and final reports will also be retained in the laboratory files and, at the discretion of the laboratory manager or QA officer, will be stored on computer disk and in hard-copy form for a minimum of five years from point of generation. Data will be made available for retrieval by authorized project staff from the designated ARDC and the laboratory archives upon request.

3. QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The overall objective of the post-decontamination characterization is to obtain data to determine if decontamination activities of Tanks WM-103 through WM-106 and WM-181 will meet the TFF closure requirements as defined by HWMA/RCRA and DOE. The DQOs are qualitative and quantitative statements derived from the first six steps of EPA's DQO process (EPA 2000) that

- Clarify the study objective
- Define the most appropriate type of data to collect
- Determine the most appropriate conditions in which to collect the data
- Specify tolerable limits on decision errors, which will be used as a basis for establishing the quantity and quality of data needed for decision-making.

3.1 Data Quality Objectives

The DQOs are discussed in the context of the DQO process as defined by the *Guidance for the Data Quality Objectives Process* (EPA 2000). EPA developed this process to ensure that the type, quantity, and quality of data used in decision-making are appropriate for the intended application. The DQO process includes seven steps, each of which has specific outputs. The DQO process has been, and will continue to be, used for each of the sampling activities conducted during the closure activities for Tanks WM-103 through WM-106 and WM-181. Each of the following subsections corresponds to a step in the DQO process, and the output for each step is provided as appropriate. Because sample collection will occur at various times during the closure activity and the data use for each sample collection activity may vary, the outputs for each DQO step will reflect these data needs and uses.

3.1.1 Problem Statement

The first step in the DQO process is to clearly state the problem to be addressed in the context of the TFF HWMA/RCRA and DOE closure activities. The intent of this step is to clearly define the problem so that the focus of the activities will be unambiguous. The appropriate outputs for this step are (1) a concise description of the problem, (2) a list of the planning team members, (3) identification of the decision-maker(s), and (4) a summary of available resources and relevant deadlines for the study. The planning team members, decision-makers, and schedule are presented in *Idaho Nuclear Technology and Engineering Center Tank Farm Facility Conceptual DOE and HWMA/RCRA Closure Approach* (INEEL 2000) and in the *Tier 1 Closure Plan for the Idaho Nuclear Technology and Engineering Center Tank Farm Facility at the INEEL* (DOE-ID 2003b). The problem statement is that there is a need to demonstrate that tank decontamination activities have met closure performance objectives.

Collected data will be used to determine if HWMA/RCRA action levels and DOE low-level waste (LLW) facility closure performance standards (25 mrem/year), which are consistent with performance standards in 10 CFR Part 61, Subpart C (2003), are met. The residue remaining in the TFF following closure cannot be characteristic hazardous waste (i.e., either characterized by toxicity or by corrosivity) (40 CFR 261, 2004). The concentration of hazardous constituents associated with the listed waste codes currently attached to the tank waste also must be below action levels. Data indicating that characteristic waste remains in any given tank, WM-181 vault sump, or cooling coil, or that concentrations of hazardous constituents are above HWMA/RCRA closure plan (DOE-ID 2004) action levels may be used

to justify additional removal and decontamination. Following the closure of all TFF tank systems, the mean concentrations for constituents remaining in the residuals in all tanks, and the WM-181 vault sump will be used to determine if the clean-closure specifications stated in the closure plan have been met or if closure to alternative DOE requirements for high-level waste facilities and/or HWMA/RCRA landfill standards will be required.

3.1.2 Decision Statement

The second step in the DQO process is to identify the decisions and the potential actions that will be affected by the data collected. This is done by specifying principal study questions and alternative actions that could result from resolution of the principal study questions, and by combining the principal study questions and alternative actions into decision statements.

The objective of the post-decontamination tank system sampling is to answer the following principal study question:

- Are post-decontamination concentrations of radioactive and hazardous constituents remaining in the TFF less than the applicable performance assessment standards and action levels specified in the HWMA/RCRA closure plan (DOE-ID 2004)?

Depending on the resolution of the principal study question, the alternative actions to be taken are as follows:

- If the concentration of any radioactive or hazardous constituent in any individual tank system component (e.g., the WM-103 through WM-106 or WM-181 tanks, cooling coils, WM-181 vault sump, and/or diversion valve boxes [DVBs]) results in a large enough contribution to the mean concentration of the constituent for the entire TFF such that an action level specified in the closure plan has been exceeded, then additional decontamination of the most contaminated tank system components will be considered.
- If the concentration of any hazardous constituent or the pH of the solution remaining in any individual tank system component (e.g., the WM-103 through WM-106 or WM-181 tanks, and/or WM-181 vault sump) results in the solution being a characteristic hazardous waste because of the toxicity or corrosivity characteristic, then the tank will undergo decontamination until the hazardous characteristic has been removed.
- If the concentrations of hazardous constituents indicate that the closure performance standards have been met, then the TFF will be closed under a HWMA/RCRA clean closure.
- If the concentrations of radionuclides indicate that the DOE Order 435.1 (2001) performance standards have been met, the tank system will be closed as a LLW landfill.
- If additional decontamination is not deemed feasible and concentrations of hazardous constituents and/or radionuclides indicate that the performance standards for the residuals in the TFF have not been met, then closure to HWMA/RCRA landfill standards or alternate requirements consistent with DOE Order 435.1 (2001) will be implemented as applicable.

Combining the principal study question and alternative actions results in the following decision statement:

- Following decontamination of the TFF tank systems, determine whether the concentrations of constituents or properties (i.e., pH) of concern in the residuals remaining in the TFF system components are below closure performance standards; if not, then HWMA/RCRA landfill standards and/or alternate DOE requirements for closure must be met.

3.1.3 Decision Inputs

The third step in the DQO process is to identify the informational inputs required to resolve the decision statement and to determine which of those inputs require measurements. The informational input needed to resolve the decision statement in Section 3.1.2 is the identification and quantification of hazardous and radioactive constituents present in each of the tank systems following decontamination.

The decision to continue or stop decontamination activities will be based on achievement of the performance standards stated in the closure plan (DOE-ID 2004) and a direct comparison to the radionuclide source term used in the *Performance Assessment for the Tank Farm Facility at the Idaho National Engineering and Environmental Laboratory* (DOE-ID 2003c). Decontamination operations for Tanks WM-103 through WM-105 and WM-181 will cease when characterization data show that (1) the mean concentrations of hazardous constituents remaining in the tanks and WM-181 vault sump are below the action levels specified in the closure plan, (2) the chromium remaining in the cooling coils of Tanks WM-103 through WM-106 is reduced to concentrations below the action level for chromium specified in the closure plan, and (3) radionuclide activities provide acceptable results when used as the PA source term or when it is determined that additional decontamination would not result in data showing these criteria are met.

Clean-closure action levels for HWMA/RCRA are defined in Section 3 of the HWMA/RCRA closure plan (DOE-ID 2004). The performance assessment (PA) (DOE-ID 2003c) used the known pre-decontamination source term; post-decontamination action levels for each radionuclide have also been established. Even if the post-decontamination characterization data show that the performance standard for an individual radionuclide has not been met, the results of the PA modeling could remain acceptable based on the reduction of other radionuclides to levels well below the action levels used in the PA. Because of this, data collected during post-decontamination characterization will be used in the PA model to determine whether decontamination has met PA action levels. All of the data collected during TFF closure operations are required before PA action levels can be assessed. Therefore, no final decisions regarding the radionuclide concentrations can be made following decontamination of only the WM-103 through WM-106 and WM-181 tanks and the WM-181 vault.

To resolve the decision statement, concentrations of the hazardous constituents and radionuclides remaining in all of the TFF tank systems must be determined. No comprehensive data pertaining to the concentrations of constituents in Tanks WM-103 through WM-106 and WM-181 are available. Through process knowledge, however, it is assumed the constituent concentrations are similar to those determined for Tanks WM-182 and WM-183. (See the *Sampling and Analysis Plan for the Post-Decontamination Characterization of the WM-182 and WM-183 Tank Residuals* [INEEL 2002] for data.) The existing data, PA source term, and key radionuclides listed in DOE Order 435.1 (2001) are relevant to this study because they provide a list of constituents for which analyses should be performed. The existing data from Tanks WM-182 and WM-183 can also be used to provide estimates of contaminant concentration variability within the tanks. Information from process knowledge of tank operations further defines the list of constituents that require analytical data after decontamination.

During this third step of the DQO process, the basis for an action level is established. The action level is the threshold value that provides the criterion for choosing among alternative actions. Action levels are derived from risk assessment methodologies. The constituent-specific action levels were

derived to ensure the protection of human health and the environment. For hazardous constituents, a description of how the action levels were derived is provided in Appendix B of the HWMA/RCRA closure plan (DOE-ID 2004). Radioactive constituents were modeled in the PA criteria to ensure the exposure to the public from residuals is less than 25 mrem/year are met (DOE-ID 2003c).

The radioactive constituents are evaluated using exposure pathways to determine appropriate clean-closure definitions. The PA presents valid conclusions that demonstrate that all pathways (air pathway, groundwater exposure pathway, and the inadvertent intruder analyses) meet the performance objectives or measures of DOE Manual 435.1-1 (2001). The PA establishes the basis for concluding the reasonable expectation of facility performance and provides reasonable assurance that the performance objectives will be met by closure of the tanks.

3.1.4 Study Boundaries

The fourth step in the DQO process is to define the spatial and temporal boundaries of the study. The spatial boundaries define the physical extent of the study area; they may be subdivided into specific areas of interest. The temporal boundaries define the duration of the entire study or specific parts of the study. The appropriate outputs of this step are a detailed description of the spatial and temporal boundaries of the problem and a discussion of any practical constraints that may interfere with the study.

The HWMA/RCRA facility closure requirements specify that the boundaries applicable to cleanup of closed facilities are the unit boundaries of the unit being closed. The boundaries for DOE high-level waste facility closures are based on the PA conducted during closure activities. For closure sampling, the TFF is divided into five general sampling locations: the tank heel residuals, the residual contents of the WM-181 vault sump, residual contents of the DVB sumps, rinsates collected from sections of waste transfer lines that have been removed from the system, and the residual contents of the decontaminated cooling coils. (The sampling and analysis of the waste transfer piping were completed in accordance with the *Sampling and Analysis Plan for the Post-Decontamination Characterization of the Process Waste Lines from INTEC Tank Farm Facility Tanks WM-182 and WM-183* [INEEL 2001].)

The media sampled to resolve the decision statement are representative portions of the rinse solutions remaining in the tanks, WM-181 vault sump, and cooling coils following decontamination activities. To characterize these residuals, samples from the various locations will be collected and analyzed. The sample analysis data of the residuals remaining in Tank WM-181 and the WM-181 vault sump will be assessed separately. The vault sample will be included with sump data collected from all five phases and compared with action levels. The mean concentration of the residuals in the WM-181 tank will be used to assess whether or not action levels were exceeded.

Decisions concerning the TFF as a whole will be made using data from several sample populations. Closure of the TFF will be based on the mean characteristics or concentrations after the waste has been removed from all TFF tank systems and decontamination has been completed. Different and separate decisions will be applied to these populations relative to continuing to clean, but the closure of the TFF as a whole is the real spatial boundary this particular sampling and analysis exercise is ultimately going to support. Similar populations will be compared using a *t*-test analysis (or other appropriate statistical method) to compare mean concentrations in the two populations. As stated earlier, the rinsates from the cooling coils in Tanks WM-103 through WM-106 will be sampled to determine the mean concentration of chromium to determine if the action level specified in the HWMA/RCRA closure plan has been exceeded. The rinsate data from the cooling coils will not be used to determine the mean concentrations of constituents in the TFF.

Defining the temporal boundaries of the problem involves specifying the timeframe the decision applies to and determining when to collect data. Closure of the TFF requires that any constituents left in place will have no adverse impacts to human health and the environment at any future date. Subsequently, decisions made at the time of closure also must apply to any future date. Because of the length of time involved, it will not be possible to collect data over this entire period. Therefore, the performance standards applied to this closure will model impacts to the environment and public radiation exposure from the tank residuals left in place. The data collected after decontamination activities are completed at each individual tank, and the WM-181 vault. This data will be combined with data from the other TFF tanks, vault sumps, and DVBS to conduct this risk assessment.

The period within which to collect the data is determined by decontamination operations; these operations will continue at any given tank, or vault until project personnel believe the decontamination is complete. At that time, one sample from the tank or vault sump may be collected and the data compared to the action levels. If the data from this initial sample are in a range where project personnel believe the required samples can meet the DQOs, the samples specified in this SAP will be collected.

In defining the study boundaries, the scale of decision-making must also be discussed. As explained previously, the performance standards will be applied to the effects of exposure to the public by leaving tank system residuals in place. Thus, to assess DOE closure requirements, the model used in the PA will drive the scale of decision-making (DOE-ID 2003c). For HWMA/RCRA closure, the decisions will apply to closure of the TFF as it is defined in the HWMA/RCRA closure plan (DOE-ID 2004). The practical constraints on data collection include the difficulties in obtaining samples from the tanks and vault sump (specifically, limited access and the potential for high-radiation fields). Several options for obtaining representative samples and/or obtaining data to characterize the contents will be investigated, including:

- Using a radiation detection device to monitor tank discharges during decontamination and to determine the appropriate times to collect samples using the simple sampler, submersible pump, or other appropriate sampling equipment. The data gathered from the beta/gamma detection equipment during decontamination of Tanks WM-103 through WM-106 and WM-181 also could be used to determine when variability of the remaining tank contents is low enough to minimize the number of samples collected in future tank decontamination activities and when to collect the initial sample discussed previously. The data from the final samples collected can be used to verify variability indicated by the radiation detection equipment.
- Collecting samples from the tanks through riser assemblies with the simple sampler, submersible pump, or other appropriate sampling equipment.
- Collecting samples of rinse solutions from the cooling coils in Tanks WM-103 through WM-106 using existing sample collection ports installed in these lines, if pressure testing confirms their integrity.
- Collecting samples of rinse solutions from removed sections of the cooling coils in Tanks WM-103 through WM-106, if pressure testing of the cooling coils determines that their integrity is uncertain.
- Collecting samples from the WM-181 vault sump using remote sampling equipment.

The sample collection option (or options) chosen will provide the most representative characterization of the sample populations while adequately protecting the health and safety of sampling team members. Limitations on data interpretation introduced by sample collection constraints

(e.g., inadequate ability to collect samples from the randomly selected sample locations and inability to collect sufficient sample volume) will be discussed in the closure activity summary reports.

3.1.5 Decision Rule

The fifth step in the DQO process is to: (1) define the parameters of interest that characterize the population, (2) specify the action level, and (3) integrate previous DQO outputs into a single statement that defines the conditions that would cause the decision-maker to choose among alternative actions. The decision rule typically takes the form of one or more “*If...then*” statements describing the action or actions to take if one or more conditions are met.

The decision rule must be specified in relation to a parameter that characterizes the population of interest. Because the tank residues will be agitated during decontamination activities, it is assumed that residual contaminants will be distributed relatively equally. It is also assumed that final rinse solutions from the cooling coil systems in Tanks WM-103 through WM-106 will be relatively homogeneous aqueous solutions with low concentrations of contaminants of concern. Therefore, the parameter of interest will be the true mean concentration of the contaminants of concern. Because it is not possible to determine the value of the true mean using sample data, a statistic must be chosen upon which the actions are based. In the case of TFF closure, the true mean will be estimated by the concentration at the 95% upper confidence limit (UCL) of the sample mean.

The decision rules are based on the HWMA/RCRA closure plan requirements that specify that no hazardous, Class C, greater than Class C, or transuranic waste may be left in place following closure and that the risks posed by the concentrations of measurable contaminants are acceptable. Therefore, the decision rules are:

- If the true mean (as estimated by the 95% UCL of the sample mean) concentration of any applicable hazardous waste constituent detected in toxicity characteristic leaching procedure (TCLP) analyses of the TFF residuals collected from any individual tank, or the WM-181 vault sump is greater than the maximum concentration of contaminants for the toxicity characteristic listed in 40 CFR 261.24 (2004), or if the true mean pH (as estimated by the 5% lower confidence limit of the sample mean for acid pH and the 95% UCL of the sample mean for basic pH) of TFF residuals collected from any individual tank or sump exhibits the characteristic of corrosivity, then either additional decontamination steps will be undertaken or closure to HWMA/RCRA landfill standards will be considered.
- If the true mean (as estimated by the 95% UCL of the sample mean) concentration of any hazardous constituent detected in total constituent analyses of the TFF residuals collected from statistically similar populations (i.e., sample locations) is greater than the action level specified in the HWMA/RCRA closure plan (DOE-ID 2004), then additional decontamination steps may be undertaken. Closure to HWMA/RCRA landfill standards will be considered at final closure of the TFF.
- If the true mean (as estimated by the 95% UCL of the sample mean) concentration for the sum of the radioisotopes in any given tank system at the time the samples are collected is not indicative of Class C waste as defined in 10 CFR 61.55 (2003), then the residual radionuclide concentration will be averaged with the mass of grout needed to enhance waste removal and stabilization.
- If the true mean (as estimated by the 95% UCL of the sample mean) concentration for the sum of the radioisotopes in any given tank system at the time the samples are collected is indicative of Class C waste as defined in 10 CFR 61.55 (2003), then, for safety and technological reasons, grout

will be added to the Class C waste to eliminate free liquids, resulting in a waste form that meets performance standards for LLW as defined in DOE Order 435.1 (2001).

- If the true mean (as estimated by the 95% UCL of the sample mean) concentration for the sum of the transuranic radioisotopes detected in the analyses of the residuals in any tank or sump is greater than 100 nCi/g, then additional waste removal and/or decontamination may be performed or the residuals will be stabilized in accordance with Chapter III of DOE Guide 435.1-1 (1999).

3.1.6 Decision Error Limits

The sixth step in the DQO process is to minimize uncertainty in the data by specifying tolerable limits on decision errors. The limits are used to establish performance goals for the data collection design. The possible range for the parameter of interest is determined, and the types of decision errors and the potential consequences of the errors are defined.

Decisions are based on measurement data; however, the data provide only an estimate of the true state of the waste. Because of this, decisions could be based on data that may not accurately reflect the true state of the TFF residuals. Therefore, if the data are not a true representation of the characteristics of the tank system residuals, the decision-maker could make a decision error. The decision-maker must define tolerable limits on the probability of making a decision error.

The probability of a decision error can be controlled by adopting a scientific approach. Using this approach, the data are used to select between the presumed condition of the decontaminated tank system residuals and the alternative condition. One of these conditions is assumed to be the baseline condition and is referred to as the *null hypothesis* (H_0). The alternative condition is referred to as the *alternative hypothesis* (H_a). The null hypothesis is presumed to be true in the absence of strong evidence to the contrary. This feature provides a way for the decision-makers to guard against making the decision error with the most undesirable consequences.

A decision error occurs when the decision-maker rejects the null hypothesis when it is true (a *false-positive decision error*) or fails to reject the null hypothesis when it is false (a *false-negative decision error*). For example, a decision-maker presumes a certain waste is hazardous (i.e., the null hypothesis is “the waste is hazardous”). However, if the data on that waste cause the decision-maker to conclude that the waste is not hazardous when it really is hazardous, then the decision-maker would make a false-positive decision error. Statisticians usually refer to this as a Type I error. The size of this error is called alpha (α), the level of significance, or the size of the critical region.

A false-negative decision error occurs when the decision-maker fails to reject the null hypothesis when it is false. In the waste example given above, the false-negative decision would be to use the data to conclude that the waste is hazardous when, in fact, it is not. Statisticians usually refer to false-negative decision errors as Type II errors. The measure of the size of this error is called beta (β); the measure is also known as the complement of the power of a hypothesis test.

The possibility of decision error cannot be eliminated; however, by controlling the total study error, it can be minimized. Methods for controlling total study error include: (a) collecting a large number of samples to control sampling design error, (b) analyzing individual samples several times, and (c) analyzing individual samples using more precise analytical methods (to control measurement error). The chosen method for reducing decision errors depends on where the largest components of total study error exist in the data set and the ease in reducing error in those data components. The amount of effort expended on controlling decision error is directly proportional to the consequences of making an error.

The two types of decision errors for the characterization of decontamination residuals for the TFF and for the WM-103 through WM-106 and WM-181 tank systems are: (a) determining that the concentration(s) of constituents in the residuals do not exceed action levels when, in fact, they do or (b) determining that the concentration(s) of constituents in the residuals exceed action levels when they, in fact, do not. The consequences of each decision error must be considered. Concluding that the residuals meet action levels when, in fact, they do not would result in the assumption that the TFF could be clean closed under HWMA/RCRA and the facility could be closed under DOE Order 435.1 (2001) action levels. The consequences of this error would be fewer controls in place to ensure protection of the public and the environment following closure when, in fact, these controls should be in place. Concluding that the residuals do not meet performance standards when, in fact, they do would result in either additional decontamination activities or proceeding with closure to HWMA/RCRA landfill standards and/or applicable DOE requirements. The consequences of this decision would be further expense of project resources to complete the additional activities, issues associated with the project schedule being unnecessarily lengthened, and the potential for generation of unnecessary waste in the form of additional rinsate solutions as further decontamination is attempted.

The decision error that has the more severe consequences as the true concentrations of the parameters of interest approach the action level must be specified. In problems that concern regulatory compliance, human health, or environmental risk, the decision error that has the most adverse consequences is established as the null hypothesis. The decision error with the more severe consequences is used because, as the parameters approach the action level, the data are much more likely to lead to an incorrect decision than when the parameters are far above or below the action level. In statistical hypothesis testing, the data must conclusively demonstrate that the null hypothesis is false, which places the burden of proof on demonstrating that the most adverse consequences will not likely occur.

Because the more severe decision error occurs when it is determined that the constituent concentrations in the tank system residuals are less than action levels when, in fact, they are not, the null hypothesis will be set as, "The concentrations of hazardous or radioactive constituents in TFF residuals following decontamination exceed action levels." The alternative hypothesis then becomes, "The concentrations of hazardous or radioactive constituents in TFF residuals following decontamination are less than action levels."

Based on these definitions of the null and alternative hypotheses, the false-positive and false-negative errors can be stated. The false-positive decision error corresponds to the more severe decision error. The false-positive error would be to conclude that the concentration of hazardous or radioactive constituents in TFF residuals following decontamination are less than action levels when, in fact, they are not. The false-negative decision error would be to conclude that the concentrations of hazardous or radioactive constituents in TFF residuals following decontamination are greater than action levels when, in fact, they are less.

A range of possible parameter values must be specified where the consequences of decision errors are relatively minor. This range of parameter values is referred to as the "gray region." The gray region is bounded on one side by the action level and on the other side by the parameter value where making a false-negative decision error begins to be significant. It is necessary to specify the gray region because the variability in the population and unavoidable imprecision in the measurement system combine to produce variability in the data such that a decision may be "too close to call" when the true parameter value is very close to the action level. In statistics, this interval is called the "minimum detectable difference" and is expressed with the Greek letter delta (Δ). The width of this gray region is a critical part of the calculation for determining the number of samples needed to satisfy the DQOs, and it represents one important aspect of the decision-maker's concern for decision errors. A narrower gray region implies a desire to detect conclusively the condition when the true parameter value is close to the action level. From a practical

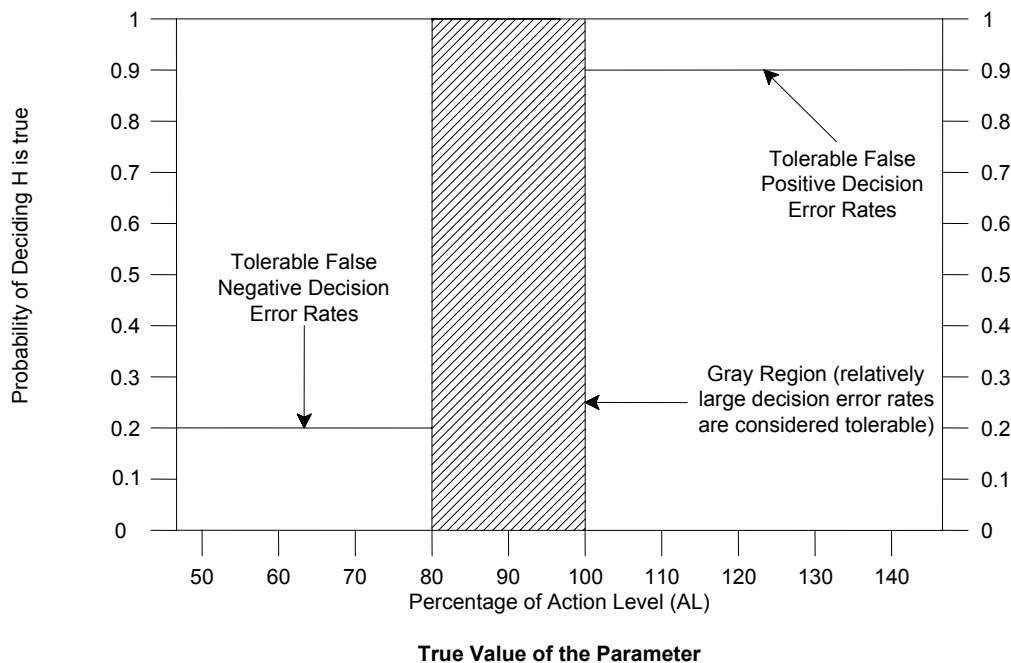
standpoint, the gray region is an area where it will not be feasible to limit the false-negative decision error rate to low levels because of high costs.

However, because the costs associated with making a false-negative decision error are relatively high for this closure activity, a narrow gray region will be appropriate. The gray region will be as follows:

- For characteristic hazardous waste determinations, the gray region will be bounded on one side by the TCLP maximum concentration for the toxicity characteristic and on the other side by a value that is 80% of the parameter-specific maximum concentration.
- For measuring the waste for the corrosivity characteristic, the gray region will be bounded on one side by a pH measurement of 2.0 (or 12.0) and on the other side by a pH measurement that deviates from the action level by 20% of the applicable action level (i.e., 2.4 or 9.6).
- For other hazardous constituents of concern in the waste, the gray region will be established between 80% and 100% of the action levels for the hazardous constituents as specified in the HWMA/RCRA closure plan (DOE-ID 2004).
- For the determination of transuranic waste, the gray region will be established between 80 and 100 nCi/g total activity for the radionuclides with an atomic number greater than 92 and a half-life greater than 10 years.
- For Class C and greater than Class C determinations, the gray region will be established between the criteria in 10 CFR 61 (2003) and 80% of these criteria.

The final activity required in specifying the tolerable limits on decision error is to assign limits to points above and below the gray region that reflect the probability of a decision error occurring. These probability values are the decision-maker's tolerable limits for making an incorrect decision when the parameter of interest (in this case, the true mean concentration) is equal to a concentration at the action limit or at the lower boundary of the gray region. Selection of the tolerable limits is done by choosing a possible true value for the parameter of interest and then choosing a probability limit based on an evaluation of the seriousness of the potential consequences of making a decision error if the true parameter value is located at that point. The EPA guidance (EPA 2000) recommends beginning the evaluation of sampling designs using 1% (a value of 0.01) as the starting point for setting decision error rates. The guidance specifies that the value of 0.01 should neither be considered a prescriptive value for setting decision error rates nor an EPA policy. Rather, it should be viewed as a starting point from which to develop decision errors applicable to the study. A graphic demonstration of these concepts is presented in Figure 1.

The project team must use the three variables (width of gray region, acceptable false-positive decision error rate when the true mean is equal to the action level, and acceptable false-negative decision error rate when the true mean is equal to the lower bound of the gray region) and adjust them to acceptable tolerances. Once this has been done, the number of samples required to satisfy the DQOs and the sample collection design can be determined.



Baseline condition: Parameter exceeds action levels.

True Concentration	Correct Decision	Type of Error	Tolerable Probability of Incorrect Decision
<80% action level	Does not exceed	F(-)	20%
80–100% action level	Does not exceed	F(-)	Gray region
>100% action level	Does exceed	F(+)	10%

Figure 1. Example of a decision performance goal diagram and corresponding decision error limits table.

3.1.7 Design Optimization

The last step in the DQO process is design optimization. The purpose of design optimization is to identify the best sampling and analysis design that satisfies all of the previous steps in the process. The activities involved in design optimization include:

- Reviewing the outputs of the first six steps and existing environmental data.
- Developing general data collection design alternatives.
- Formulating a mathematical expression to solve the design problem for each data collection design alternative. The mathematical expression that resolves the design problem takes into account things like known skewness of the data (or some other known attribute that causes the data to be other than normally distributed). The expression then is used to determine the appropriate number of samples to collect to have a known possibility of error in decision-making once the data are collected.

- Selecting the optimal number of samples to satisfy the DQOs for each data collection design alternative.
- Selecting the most resource-effective data collection design that satisfies all the DQOs.

After these activities are completed, the operational details and theoretical assumption of the selected design are documented in the SAP.

The outputs of the first five steps have been discussed previously. Environmental data are available for the Tanks WM-182 and WM-183 system contents before and after decontamination activities. However, these data only can be used to assume information concerning the possible range of concentrations (or values) that will be measured for the constituents (and property) of interest following decontamination of Tanks WM-103 through WM-106 and WM-181. Because no comprehensive environmental characterization data exist for the contents of Tanks WM-103 through WM-106 and WM-181, only assumptions of the parameter variability and possible concentration ranges can be made based on available data from Phase I closure activities conducted at Tanks WM-182 and WM-183.

Because of the difficulty in obtaining samples from the tanks, data collection design alternatives are limited. The planning assumptions for the project include some assumptions related to sample collection. Specifically, the assumption has been made that representative samples of post-decontamination residuals in the tanks, and the WM-181 vault sump can be collected through the riser assemblies using the simple sampler, submersible pump, or other grab sampling techniques. It also is assumed that if only a liquid phase is obtained using these techniques, the solid phase is assumed inconsequential and can be ignored. If solid phase is obtained, it will be segregated and analyzed separately by the laboratory. For the residuals in the tank heel, if solids exceed 15% by volume of the total sample collected at a given location (see discussion in Section 5.1.2), the solids will be separated and analyzed.

For the WM-181 vault sump, the total sample volume needed for analysis would consume most of the sump's contents. Because of the volume of sample collected relative to the total volume of the sampled population, one sample will give a very good estimate of the true mean concentrations for constituents of concern. It is estimated that the vault sump will contain only a small volume of rinsate following decontamination activities. Furthermore, the vault sump contains piping that limits access and makes it difficult to collect large volumes of sample. Therefore, only one sample will be collected for the required analyses from the sump in the WM-181 vault and the selected DVB sumps. Because all of the cooling coils in Tanks WM-103 through WM-106 can be sampled, a random sample design can be applied. The process used to sample and analyze the waste transfer lines was documented in the *Sampling and Analysis Plan for the Post-Decontamination Characterization of the Process Waste Lines from INTEC Tank Farm Facility Tanks WM-182 and WM-183* (INEEL 2001).

Tank samples can only be collected through the risers. Because the simple sampler and submersible pump have no reach away from the location directly beneath the risers through which they are lowered, it is not possible to have a truly random sampling design where all locations in the bottom of the tank have an equal opportunity of being sampled. Because the volume of residual liquid remaining in the system will be greatest in the tanks, the greatest potential for risk from the presence of constituents following decontamination exists in the tanks. Consequently, it is important to apply a very defensible sampling design to the post-decontamination residuals in the tank heels.

For the application of statistical hypothesis testing, the sampling design will be simple random sampling. Despite the limitations described, a simple random sampling design can be applied to the tank residuals. In simple random sampling, every point in the population has an equal chance of being

selected. Simple random sample designs are chosen when the variability of the medium is relatively small and sufficient resources are available to conduct the required number of analyses. Because the sampled populations will be agitated during decontamination activities, the possibility of “every point in the sample medium having an equal opportunity of being selected” is valid. The assumption of random sampling statistics is, therefore, valid for characterization of the liquid phase present in the tanks and for rinsate solutions following decontamination of the cooling coils. These solutions will be aqueous-based liquids, which tend to be relatively homogeneous (assuming there is no solids content in the samples collected). Therefore, the sample collection points accessible through the risers, vault sump, DVB access ports, and cooling coils are just as likely to obtain a random sample as any other sample collection point.

In the case of the WM-181 vault sump and DVB sumps, statistical hypothesis testing will not be applied because the samples will represent such a large proportion of the entire population, and the analysis results will be very close to any sample mean that would be determined if multiple analyses were possible. That is, the values obtained from single measurements from samples of these locations will be used.

The sampling design that will be used for the tank residuals and cooling coils is stratified random sampling. Stratified random sampling uses a random sampling approach within each of the two strata (i.e., tanks and cooling coil sets). Two data collection design alternatives can be followed for using a random sampling approach within sample location types: simple random sampling or composite random sampling. In simple random sampling, several locations are randomly chosen and separate samples are collected and analyzed from each. In composite random sampling, multiple samples are collected and physically combined (composited) and one or more sub-samples are drawn for analysis. Because of the nature of the sample collection logistics and personnel safety concerns, composite sampling will not be an acceptable alternative for sampling the TFF tank system components. Therefore, the option of composite random sampling will not be considered further.

Another sampling approach to be considered is systematic sampling, which is usually the method of choice when estimating trends or patterns of contaminants over space or time. Systematic sampling also is useful in estimating the mean concentration when trends and patterns in concentration are not present or are known a priori, or when strictly random methods are impractical. In systematic sampling, samples are taken at locations and/or times according to a spatial or temporal pattern (for example, at equidistant intervals along a line or within a grid pattern). The inaccessibility of some portions of the tanks would make this approach difficult to implement in this activity. However, the use of a temporally systematic approach may be beneficial for the real-time radiation measurements taken as the decontamination activities proceed.

Commonly accepted mathematical expressions are used to solve the design problems for a simple random sampling approach. A mathematical expression is used to test the statistical hypothesis and define the formula for determining the number of samples required with the chosen design alternative. In some cases, a reliable estimate of the population variance is not available for determining the number of samples. This activity presents such a case. However, in such cases, an estimate of the relative standard deviation (coefficient of variation) is used. The approach is to use the relative error formula, Equation (1), to solve for error probability as shown in Equation (2).

$$d_r = \frac{|\bar{x} - \mu|}{\mu} \quad (1)$$

$$Prob(|\bar{x} - \mu| \geq d_r \mu) = \alpha \quad (2)$$

where

- d_r = relative error
- \bar{x} = sample mean
- μ = population mean
- α = false-positive value.

The formula for computing the number of samples required for a simple random sampling approach is shown in Equation (3).

$$n = \left(\frac{Z_{1-\alpha/2} \eta}{d_r} \right)^2 \quad (3)$$

where

- n = number of samples required
- Z_p = the p^{th} percentile of the standard normal distribution (from statistical tables)
- η = coefficient of variation or σ/μ
- d_r = relative error or the absolute value of the difference of the sample mean and population mean, which is then divided by the population mean
- σ = population standard deviation
- μ = population mean.

Because it is assumed that the variability of the liquid matrix will be low (it will be relatively homogeneous throughout the volume remaining in the tank and cooling coils), a low coefficient of variation can be chosen. Therefore, a coefficient of variation of 20 is used, and the assumption is made that it is acceptable to have a 10% chance of getting a set of data for which the relative error exceeds 15%. Hence, $Z_{1-0.10/2} = 1.645$, $\eta = 0.20$, and $d_r = 0.15$. An example of how the number of samples is derived using these variables is given in Equation (4).

$$n = \left[\frac{1.645(0.20)}{0.15} \right]^2 = 4.8 \quad (4)$$

Therefore, five samples from the liquid matrix will be needed to meet the project DQOs.

Solid material recovered during sampling is likely to be more variable. The number of solid samples should be increased using an assumption of this variability. If it is accepted that agitation of the solids during decontamination activities will result in the solids being as homogeneous as the liquid matrix, then five samples would suffice in meeting the project DQOs for this matrix also.

Another method for calculating the appropriate number of samples to collect uses estimates for the variability of the sampled matrices, acceptable decision error rates, and the width of the gray region. To meet these DQO requirements, the number of samples required for each analyte must be determined

before sampling takes place. It is assumed that the samples will be taken via simple random sampling. To calculate the number of samples to collect (i.e., the sample size), the following must be known:

- Size of the minimum detectable region (Δ)
- Standard deviation of the concentration of the analyte (σ)
- Chance of making a false-positive decision error (α)
- Chance of making a false-negative decision error (β).

These quantities are defined in Section 3.1.6.

It is assumed that a minimum detectable difference, or gray region, for the TFF sampling that is bounded by the action level on one side and 80% of the action level on the other side will be acceptable. Using this assumption, $\Delta = 0.20$. It is not known what the standard deviation (σ) will be for any constituent once the tanks are cleaned. In the post-decontamination liquids collected from Tank WM-182, the largest standard deviation observed (as a percentage of the action level) was for mercury, measured at 12.5%. Therefore, an estimate of 12.5% of the action level will be used to calculate n for this sampling effort. Given these values for Δ and σ , the sample size can be calculated with various values for α and β . Table 3 provides the sample size estimates for various values of the chance of false-positive error (α) and the chance of false-negative error (β). Assuming a simple random sample is being taken, the formula to calculate the sample size is shown in Equation (5).

$$n = \frac{(z_{1-\alpha} + z_{1-\beta})^2 \sigma^2}{\Delta^2} + \frac{1}{2} z_{1-\alpha}^2 \quad (5)$$

where

- z_x = the x^{th} quartile of the standard normal distribution
- α = false-positive rate
- β = false-negative rate
- σ = estimated standard deviation of the population
- Δ = minimum detectable difference.

Table 3. Required sample size (n) associated with various false-positive error rates (α) and false-negative error rates (β) when $\sigma = 12.5\%$ of the action level and the width of the gray region is from 80% to 100% of the action level (i.e., $\Delta = 0.20$).

α	β	n	α	β	n
0.01	0.01	11.16	0.15	0.01	4.95
	0.05	8.87		0.05	3.35
	0.10	7.79		0.10	2.64
	0.15	7.12		0.15	2.22
	0.20	6.63		0.20	1.91
	0.25	6.22		0.25	1.68
	0.30	5.88		0.30	1.49
	0.35	5.58		0.35	1.33
0.05	0.01	7.51	0.20	0.01	4.27
	0.05	5.58		0.05	2.77
	0.10	4.70		0.10	2.12
	0.15	4.16		0.15	1.73
	0.20	3.77		0.20	1.46
	0.25	3.45		0.25	1.25
	0.30	3.19		0.30	1.08
	0.35	2.96		0.35	0.94
0.10	0.01	5.91	0.25	0.01	3.61
	0.05	4.17		0.05	2.41
	0.10	3.39		0.10	1.91
	0.15	2.92		0.15	1.58
	0.20	2.58		0.20	1.33
	0.25	2.32		0.25	1.14
	0.30	2.10		0.30	1.00
	0.35	1.91		0.35	0.89

Using Equation (5) and Table 3, it becomes apparent that if the assumption concerning data variability holds (i.e., $\sigma \leq 12.5\%$ of the action level for all constituents of concern), the sample size proposed (i.e., five samples) will allow an assessment of the null hypothesis with a 5% false-positive decision error rate and a 10% false-negative decision error rate. Therefore, for this sampling activity, a sample size of five will be used for the tank heels. Also, five samples will be taken from the cooling coils in Tanks WM-103 through WM-106 to estimate the mean concentration of chromium remaining in all of the coils.

3.2 Data Quality

The data generated from the post-decontamination characterization effort for the WM-103 through WM-106 and WM-181 tank systems will be used to evaluate parameters that are pertinent to the closure process. Each parameter to be evaluated requires data of specific quality. To demonstrate compliance with the closure requirements, the chemical and radiochemical measurement data obtained must be of high quality. Laboratory analytical procedures and laboratory data reporting will adhere to the following QA/QC standards with minor modifications:

- SW-846 methods (EPA 1998) with ER-SOW-394 (2002) standard plus raw data deliverable reporting requirements.

No modifications to the requirements for radionuclide analyses specified in ER-SOW-394 will be required. For chemical analysis, the SW-846 methods will be followed as published except as modified by ER-SOW-394. The ER-SOW-394 document imposes additional QC, including corrective actions, if a QC parameter is not within control limits. These requirements are more explicit than the published SW-846 methods and provide a more consistent data set for INEEL data users. The INEEL ER-SOW-394 requires that the SW-846 method be performed as published (with specific QC requirements) unless modifications are required because of the radioactivity of the sample. It is anticipated that the residuals and rinsates will have a low enough radioactivity to allow normal processing of the sample. However, if the sample has higher radioactivity, smaller sample aliquots may be required to protect the health and safety of laboratory personnel.

It is possible that sample volumes smaller than those required by SW-846 methods may be collected from the tanks, or the WM-181 vault sump because of limitations with sampling equipment. If this is the case, the detection sensitivity for the analytical methods will be adjusted. That is, a smaller aliquot results in a higher detection limit. In all cases where the sample aliquot does not meet SW-846 method requirements, the laboratory will document the deviation and any changes in the method in the sample analysis narrative provided with the data. The laboratory staff and their experience will be relied upon, in conjunction with the PM and PQAO, to make the best decisions for analyses where deviations may arise.

Tables 4 and 5 provide a summary of all analyses planned for the post-decontamination sampling effort of the WM-103 through WM-106 and WM-181 tank heels. The tables include the corresponding analytical method requirements for each analysis and the reporting procedure requirements. The laboratory will flag nonconforming data as appropriate and as required by the analytical laboratory statement of work (SOW).

Table 4. Summary of analysis requirements for solid residuals remaining in Tank WM-103 through WM-106 and WM-181 system components following decontamination.

Requested Analysis for Tank WM-103 through WM-106 and WM-181 Solids ^a	Analysis Method ^a	Reporting Requirements
TCLP Analysis As, Ba, Cd, Cr, Pb, Hg, Se, Ag	SW-846 1311 TCLP Extraction 3010A Sample Preparation (all elements except Hg) 6010B 7470A Hg	ER-SOW-394 Standard plus raw data deliverable
Total Metals Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Tl, V, Zn	SW-846 6010B 7471A Hg CVAA 7060A As GFAA 7740 Se GFAA INEEL ACMM 8108 Total Dissolution Sample Preparation	ER-SOW-394 Standard plus raw data deliverable
Radiochemical Parameters ^b ²⁴¹ Am, ¹⁴ C, ⁶⁰ Co, ¹³⁴ , ¹³⁷ Cs, ¹²⁹ I, ²³⁷ Np, ⁶³ Ni, ⁹⁰ Sr, ⁹⁹ Tc, ⁹⁴ Nb, ¹⁵⁴ , ¹⁵⁵ Eu, ²⁴⁴ Cm, ²³⁸ , ^{239/240} , ²⁴¹ Pu, ²³⁴ , ²³⁵ , ²³⁶ , ²³⁸ U	ER-SOW-394	ER-SOW-394 Standard plus raw data deliverable
Organic Analyses VOA, SVOA, Methanol, and PCBs	SW-846 8260B VOA 8270C SVOA 8015B Methanol by Direct Injection 8082 PCBs	ER-SOW-394 Standard plus raw data deliverable

a. ACMM = Analytical Chemistry Methods Manual (INEEL 2003)

CVAA = Cold vapor atomic adsorption

GFAA = Graphite furnace atomic adsorption

PCB = Polychlorinated biphenyl

SVOA = Semivolatile organic analysis

VOA = Volatile organic analysis.

b. This list includes those key radionuclides that contribute significantly to the PA (DOE-ID 2003c), have readily available methods of analysis, and are described in DOE Manual 435.1-1, Chapter II (2001).

Table 5. Summary of analysis requirements for liquid residuals remaining in Tank WM-103 through WM-106 and WM-181 system components following decontamination.

Requested Analysis for Tank WM-103 through WM-106 and WM-181 Liquids ^a	Analysis Method ^a	Reporting Requirements
Total Metals Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Tl, V, Zn	SW-846 3010A Sample Preparation (all elements except Hg) 6010B (all elements except Hg) 7470A Hg	ER-SOW-394 Standard plus raw data deliverable
Radiochemical Parameters ^b ²⁴¹ Am, ¹⁴ C, ⁶⁰ Co, ¹³⁴ , ¹³⁷ Cs, ³ H, ¹²⁹ I, ²³⁷ Np, ⁶³ Ni, ⁹⁰ Sr, ⁹⁹ Tc, ⁹⁴ Nb, ¹⁵⁴ , ¹⁵⁵ Eu, ²⁴⁴ Cm, ²³⁸ , ^{239/240} , ²⁴¹ Pu, ²³⁴ , ²³⁵ , ²³⁶ , ²³⁸ U	ER-SOW-394	ER-SOW-394 Standard plus raw data deliverable
Wet Chemical Parameters Chloride, Fluoride, Nitrate, Phosphate, Sulfate	SW-846 9056	ER-SOW-394 Standard plus raw data deliverable
pH	9040B or 9045C	
Organic Analyses VOA, Methanol and SVOA, PCBs	SW-846 8260B VOA 8270C SVOA 8015B Methanol by Direct Injection 8082 PCBs	ER-SOW-394 Standard plus raw data deliverable

a. PCB = Polychlorinated biphenyl
SVOA = Semivolatile organic analysis
VOA = Volatile organic analysis.

b. This list includes those key radionuclides that contribute significantly to the PA (DOE-ID 2003c), have readily available methods of analysis, and are described in DOE Manual 435.1-1, Chapter II (2001).

4. DOCUMENTATION AND DATA MANAGEMENT

Documentation involves the recording of all events relating to field and laboratory activities. Typical field documentation will include field logbooks, sample labels, and COC forms. Sample handling procedures include COC, radiological field screening, sample- and investigation-derived waste packaging, and transport of samples to the laboratory.

4.1 Documentation

To ensure that all sampling, analysis, and data reporting activities are conducted in accordance with project DQOs and all appropriate safety procedures, adequate documentation of each event must be completed. Therefore, all field activities related to sample collection, site safety, and sample custody must be recorded by the FTL or designee. In addition, all laboratory activities relating to sample custody, sample preparation, sample analysis, and data reporting must be recorded to ensure that laboratory data can be confidently assigned to field sample points. The PE will observe sampling activities and will be given the logbooks, COC forms, analytical results, and any other documentation generated during closure activities that is required to certify the closure.

The laboratory will perform all functions required for Tanks WM-103 through WM-106 and WM-181 samples in accordance with an approved laboratory QA plan. The PM and other key project staff may contact the laboratory personnel and obtain a copy of the laboratory QA plan and/or visit the facility to ensure that laboratory procedures meet the project-specific goals.

4.1.1 Field Operations Records

The following subsections provide a summary of requirements for adequate field documentation. All field documentation, document control, and daily updating of field logbooks and field materials will be the responsibility of the FTL or designee.

4.1.1.1 Sample Container Labels. At the point of sample collection, samples will be collected in pre-labeled, 1-L containers to obtain sufficient volumes for the required analyses. These containers immediately will be transferred to the INTEC Remote Analytical Laboratory (RAL), where the samples will be split or shielded and preserved according to volume and SAP requirements. A COC form will be generated to accompany the samples to the RAL. The RAL personnel will keep a record of the sample splits, preservation methods, and filtering performed. Sample identification numbers will be assigned to each sample split. Additionally, each split sample will be appropriately labeled and a new COC form will be generated to accompany the sample splits. Copies of the original COC and the apportionment record may accompany the new COC to establish a complete record of the sample history.

If attaching labels is difficult because of high radiation fields, the sample will be tracked using COC forms and/or entries into the project logbook. The following four entries are required to track samples appropriately:

1. Project name
2. Name or initials of sampling team member
3. Analysis request(s)
4. Field identification number.

NOTE: The time and date of sample collection will be recorded in the field logbook and/or on the COC form.

This procedure will be repeated each time a sampling team member draws a sample from Tanks WM-103 through WM-106 and WM-181 with the simple sampler, submersible pump, or other appropriate sampling equipment.

Following transport of the sample to the RAL, the RAL sample custodian will retain custody of the samples. The RAL will be tasked with segregating the liquid phase of the sample from the residual solids and separating aliquots of each sample as follows:

- A portion shall be placed in container(s) for organic analysis, taking care to minimize aeration of the sub-sample, and the sub-sample shall be preserved as soon as possible
- For all other requested analytes, the lab will separate liquids and solids by filtering, as necessary, into separate sample containers based on the volume or mass necessary for each analysis or analysis type required.

Samples and sample splits will be labeled, recorded, and tracked according to the requirements of this SAP. The following specific information will be placed on the sample label for each media type and each split of the bulk sample, and will be recorded on the COC or internal tracking forms:

- Project name
- Date of sample collection
- Time of sample collection
- Analysis request(s)
- Field identification number.

Each sample will be assigned a unique identification number. A systematic character identification code will be used to identify the samples. Uniqueness is required to maintain consistency. A field identification number will be assigned when the bulk sample is pulled. A two-character suffix will be added to the field identification number and assigned to each sample split to differentiate between individual aliquots.

4.1.1.2 Field Sampling Logbooks. Field logbooks are legal documents; they are the written record for all field data gathered, field observations, field equipment calibrations, samples collected for laboratory analysis, and sample custody. The logbooks are maintained to ensure that field activities are properly documented and that site safety meetings and site work are conducted in accordance with health and safety procedures. Field logbooks will be bound and will contain consecutively numbered pages. All entries in field logbooks will be made using permanent ink pens or markers. All mistakes made as entries will be amended by drawing a single line through the entry. The person making the correction will initial and date it. At a minimum, the following entries will be made to the field logbook:

- Identification of all sampling team members
- References to field methods used to obtain samples, field data, etc.

- Location and description of each sampling point
- Types, numbers, and volumes of samples (when observable)
- Dates of sample collection, times of sample collection, and sample identification numbers
- Dates and times of sample shipping or transfer of sample custody
- Observed weather conditions
- All field measurements
- Any deviations from the standard or expected procedure
- The COC form numbers and copies of the COC forms.

4.1.1.3 Chain of Custody Record. COC procedures will begin immediately after collection of the first sample. At the time of sample collection, the sampling team will initiate a COC form for each sample. All samples collected will then remain in the custody of a sampling team member until custody is transferred to the laboratory sample custodian. Upon receipt at the laboratory, the sample custodian will review sample labels and the COC form to ensure completeness and accuracy. If discrepancies are noted during this review, immediate corrective action will be sought with the sampling team member(s) identified on the COC as delivering the samples. If errors cannot be corrected with the sampling team members, the sample custodian will seek the PQAO or PM to correct sample labeling or COC errors.

Pending successful corrective action, the laboratory sample custodian will sign and date the COC form, signifying acceptance of delivery and custody of the samples. The sampling team will retain a copy of the signed COC and will note the time of sample custody transfer in the field logbook. Sufficient copies of COCs will be made at the time of sample delivery to ensure that appropriate personnel have copies. The laboratory will maintain possession of the original COC form until completion of sample analysis and will maintain COC copies for the term of data storage at the laboratory. The original COC form will leave the laboratory's control only when the laboratory data are disposed of or when the data are transferred to the project ARDC. The original COC form will be returned to the project file maintained by the PM or the PQAO along with the final data package deliverable.

In the field, samples will be collected in 1-L containers, which will be transported to the INTEC RAL so the appropriate sample aliquots can be prepared. Because of the potential for solid separations and the need to perform sample splitting for various analyses, the RAL will generate a sample apportionment and compositing record and various internal aliquot-tracking records at the time of sample aliquot/split handling. This record will allow the samples to be clearly tracked when portions of the original sample become segregated and/or composited before shipment to the analytical laboratory performing the required analyses. Specific information that will appear on each internal tracking record for a sample or group of samples will include:

- Sample numbers specific to sample location and media (i.e., field identification number)
- The unique sample identification number assigned to each aliquot obtained from the original field sample
- The printed form and signatures of sampling team members handling the sample

- Dates and times of aliquot/split preparation for each sample (the time entry is necessary only if the holding time is two days or less)
- Signature of any person who has maintained sample custody for any period
- Dates and times of sample possession for each person holding sample custody (the time entry is necessary only when the holding time is two days or less)
- Analyses requested for each sample and each phase
- Number of bottles of each sample.

If a laboratory other than the RAL will be performing the analyses on the sample aliquots, a new COC form will be prepared showing the sample identification numbers for the various aliquots and the requested analyses. The laboratory person responsible for preparing the sample aliquots will be listed on the COC. The RAL sample custodian will then sign the form indicating relinquishment of custody before receipt by the analytical laboratory performing the analyses. This new COC form will be transferred with the sample aliquots to the analytical laboratory performing the analyses, signed by the laboratory sample custodian, and a copy returned to the COC records coordinator identified in the analytical laboratory SOW. Copies of the internal tracking record will be retained by the RAL, PQAO, and PM, and submitted to the project ARDC.

4.1.2 Laboratory Records

Laboratory records are required to document all activities involved in sample receipt, processing, analysis, and data reporting. The following sections describe the laboratory records that will be generated for this project.

4.1.2.1 Sample Data. These records contain the times that samples were analyzed to verify that the holding times were the same as those prescribed by the analytical methods. Sample data records shall include information on the total number of samples analyzed in a given day, location of sample analysis (i.e., instrument identification number), any deviations from analysis standard operating procedures and/or methods, and time and date of analysis. Corrective action steps taken to rectify situations that did not conform to laboratory standard operating procedures and/or analytical methods (including steps taken to seek additional sample material if required) also should be noted in these records.

4.1.2.2 Sample Management Records. Sample management records document sample receipt, handling and storage, and date of analyses. The records verify that the COC was maintained and the sample was properly preserved. The record shall reflect any anomalies in the samples (such as receipt of damaged samples), note proper log in of samples into the laboratory, and address procedures used to prioritize received samples to ensure that holding time requirements will be met.

4.1.2.3 Test Methods. This documentation describes any deviation from the analytical methods or laboratory standard operating procedures. Items to be documented include sample preparation and analysis, instrument standardization, detection and reporting limits, and test-specific QC criteria. Documentation demonstrating laboratory proficiency with each method used should also be included in this category.

4.1.2.4 Quality Assurance/Quality Control Reports. QA/QC reports will include general QC records, such as initial demonstration of the capability of individual analysts to conduct specific analyses, instrument calibration, routine monitoring of analytical performance (e.g., control charts), and calibration

verification. Project-specific information from the QA/QC checks, such as blanks (e.g., field, reagent, and method), spikes (e.g., matrix, matrix spike duplicate, and surrogate), calibration check samples (e.g., zero check, span check, and mid-range check), replicates, and splits should be included in the QA/QC reports to facilitate data quality analysis. Specific requirements for the quantity and types of QA/QC monitoring and associated reporting formats will be specified in the analytical SOW to the laboratory.

4.2 Document Control

Document control consists of the clear identification of all project-specific documents in an orderly form, secure storage of all project information, and controlled distribution of all project information. Document control ensures controlled documents of all types related to the project will receive appropriate levels of review, comment, and revision as necessary. It also ensures that all documents that will ultimately affect project QA are correct before use.

The PM is responsible for properly maintaining active project files. Upon completion of the WM-103 through WM-106 and WM-181 post-decontamination tank system characterization, the PM will transfer all hard-copy information and documentation developed from the project to the designated ARDC for appropriate archiving. Hard-copy information and documentation include field logbooks, field and laboratory COC forms, laboratory reports and data, engineering calculations and drawings, final design reports, and all other technical reports related to the project. Copies of all analytical data and final reports will also be retained in the laboratory files and, at the discretion of the laboratory manager or QA officer, will be stored on computer disk and in hard-copy form for a minimum of five years from point of generation. Data will be made available for retrieval by authorized project staff from the designated ARDC and the laboratory archives upon request.

4.3 Data Management

Data management consists of controlling the data generated and other data collected (e.g., existing data) for use during this sampling and analysis effort. All data will be controlled using the document control processes described in Section 4.2 and in accordance with all existing MCPs concerning control and archival of electronic data. Data will be made available for retrieval by authorized project staff from the designated ARDC and the laboratory archives upon request.

5. SAMPLING PROCESS DESIGN

Sample handling for the post-decontamination characterization of Tanks WM-103 through WM-106 and WM-181 will require a series of special procedures because of the potential to encounter high-radiation fields in the samples and the potentially high levels of other hazardous constituents. The following subsections outline the specific sampling process design for this effort.

5.1 Sample Collection

The overall purpose of the post-decontamination sampling and analysis effort for Tanks WM-103 through WM-106 and WM-181 is to provide data to determine if the TFF decontamination activities have resulted in the HWMA/RCRA closure standards and DOE Order 435.1 (2001) requirements being met. The HWMA/RCRA performance standards include demonstrating that no hazardous waste remains in the closed unit (i.e., the TFF) and incorporating the risk-based approach to clean closure of RCRA units. The recommended risk from carcinogens is 10^{-4} using EPA default exposure parameters and a hazard quotient of 1 for noncarcinogens (EPA 2003). DOE closure requirements are based on the PA criteria found in DOE Order 435.1 (2001).

Samples of the WM-103 through WM-106 and WM-181 post-decontamination tank system residuals must be collected and analyzed for a specific group of parameters to satisfy HWMA/RCRA and DOE requirements for TFF site closure. Previous sampling efforts undertaken during process operations and in the initial characterization sampling have yielded some process-specific data. However, these data only pertain to liquids present in the tank at the time of sampling; therefore, the post-decontamination residuals must be sampled.

5.1.1 Pre-Sampling Meeting

Before sampling takes place, project personnel will meet to ensure that the sampling and analysis can be performed in a safe manner and will provide usable data. Project management personnel, sampling team members, and health and safety personnel are required to attend the pre-sampling meeting. Other project personnel may attend, as necessary.

Sampling team members must be experienced in operation of the simple sampler, submersible pump, or other appropriate sampling equipment and other aspects of sampling the TFF tanks. They will be trained in the procedures for operation of the chosen sampling equipment as well as appropriate INTEC and INEEL ESH&Q procedures and policies. The senior personnel will be familiar with the TFF systems and components.

5.1.2 Sample Location and Frequency

The nature of the sample matrix and the method of collection may place limitations on sampling and analysis design. For example, the samples from the tanks that are collected with a simple sampler or a submersible pump device will require additional sample screening and manipulation activities in the field or in the RAL before the sample is transported to the analytical laboratory.

For Tanks WM-103 through WM-106, each tank will be sampled using the simple sampler or submersible pump to collect one sample from each of the four risers (see Figure 2). The submersible pumps will then be raised, the tank contents agitated again, the pumps lowered, and a fifth sample collected from a riser chosen randomly at the time of sample collection. Tank WM-181 contains five risers; however, one riser (TR-18) contains the steam jet, leaving only four accessible for sampling. For Tank WM-181, the simple sampler or submersible pump will be used to collect one sample from each of

the four risers (see Figure 3). The submersible pumps will then be raised, the tank contents agitated again, the pumps lowered, and a fifth sample collected from a riser chosen randomly at the time of sample collection. This will result in a total of five samples from each tank. Samples may be collected using appropriate sampling equipment other than the simple sampler or submersible pump.

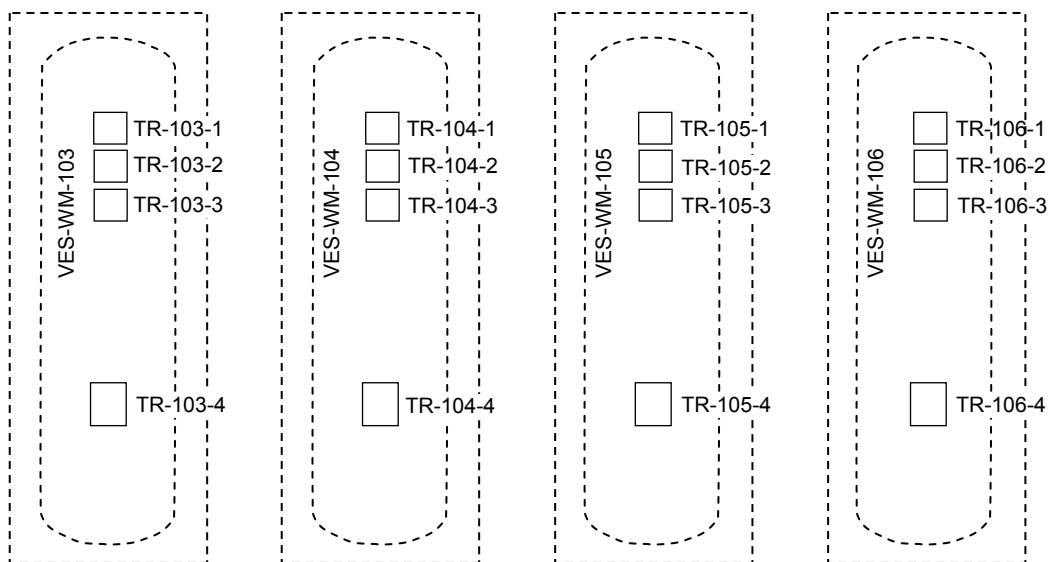


Figure 2. Riser locations where samples will be collected from WM-103, WM-104, WM-105, and WM-106.

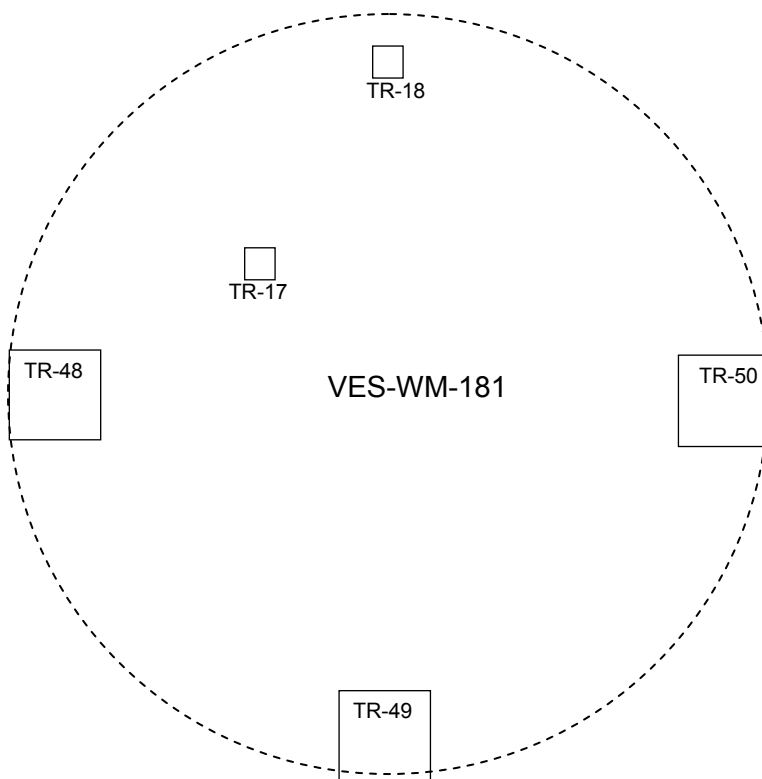


Figure 3. Riser locations where samples will be collected from WM-181.

The sample volume extracted by the submersible pump is limited only by the capacity of the receiving vessel. The maximum amount of sample volume possible (up to the total amount required for the specified analyses) will be collected each time the submersible pump is lowered into the tank.

The sample chamber of the simple sampler has a collection capacity of approximately 250 or 500 mL. It may require several trips into the tank to obtain the required volume necessary for analysis. The maximum amount of sample volume possible will be collected each time the simple sampler is lowered into the tank.

Because of the likelihood of a small sample volume and the low percentage of solids in the sample matrix, collecting a sufficient amount of solid to perform all of the required analyses may be difficult. The SAP assumes that decontamination will be complete enough so that any solids remaining in the tank will be considered residuals for which characterization is not required. If less than 15% of the sample volume recovered is solid, the assumption will be made that the solid fraction is inconsequential to decisions relative to closure.

When less than 15% of the total volume of the sample collected is solid material (i.e., less than 15% of the total volume required for all analyses), the solid fraction will be filtered at the analytical laboratory (after sample aliquots are collected for volatile organic compound [VOC] analyses) and used for as many radionuclide analyses as possible. Solid samples are included in the previous and remaining sections of the SAP only for the contingency that solids are present at greater than 15% of the total volume of the samples collected from the tanks. Following decontamination, if greater than 15% of the volume of a sample collected is solids (i.e., greater than 1.2 L collected at a single sample location), a decision will be made whether to continue or stop decontaminating the tanks to attempt to reduce the solids content. As samples are collected, the percentage of solids in each sample collection container will be measured.

The simple sampler or submersible pump can only reach the residual directly beneath the riser through which it is lowered. However, because the residual will be agitated before sampling, it is reasonable to assume that the liquid in the tank is homogeneous. Therefore, even though sample locations cannot be selected at random, potential samples are randomly distributed throughout the tank. Thus, a simple random sampling method can be assumed for sampling with the simple sampler or the submersible pump.

Enough sample material will be collected using the selected sampling method to fill the number of bottles required to complete the analyses (see Tables 6 and 7). The samples will be inspected for color, light transmission, wet weight of solids, and other qualitative indicators when they are transferred to the RAL for sample apportioning. These sample characteristics will be recorded by the sampling team members to document the completeness of removal of the former tank heel. When the sample is removed from the tank, radiation fields will be measured and recorded in the sampling logbook.

If repeated decontamination attempts fail to reduce the volume of solids collected in the samples to less than 15% of the total volume, the solid portion of the sample will be analyzed for hazardous constituents. Regardless of the relative volume of solid material present in the sample, a sample of the solids must be analyzed for radionuclides to provide data for decisions relative to DOE Tier 1 closure (DOE-ID 2003b).

Table 6. Summary of sample collection, holding time, and preservation requirements for radiological analyses.

Analysis	Sample Matrix	Approximate Volume ^a (L)	Container Type	Holding Time ^c	Preservative
Alpha Spectrometry					
Americium (²⁴¹ Am)	Water	1	HDPE ^b	≤6 months	HNO ₃ to pH < 2
Curium Isotope (²⁴⁴ Cm)	Water	1–2	HDPE	≤6 months	HNO ₃ to pH < 2
Neptunium (²³⁷ Np)	Water	1	HDPE	≤6 months	HNO ₃ to pH < 2
Plutonium Isotopes (^{238, 239/240, 241} Pu)	Water	1	HDPE	≤6 months	HNO ₃ to pH < 2
Uranium Isotopes (^{234, 235, 236, 238} U)	Water	1	HDPE	≤6 months	HNO ₃ to pH < 2
Gamma Spectrometry					
Project-specific Target Analytes (⁶⁰ Co, ¹³⁴ Cs, ¹³⁷ Cs, ⁹⁴ Nb, ^{154, 155} Eu)	Water	0.5–2	HDPE	≤6 months	HNO ₃ to pH < 2
Gamma Spectroscopy and α/β Radiochemistry	Residual Solids	16 oz	Wide-mouth plastic jar	≤6 months	None
Specific Analysis					
Carbon (¹⁴ C)	Water	0.3–1	HDPE	≤6 months	None
Iodine (¹²⁹ I)	Water	1	Amber-colored Glass ^d	≤6 months	None
Nickel (⁶³ Ni)	Water	0.5–1	HDPE	≤6 months	HNO ₃ to pH < 2
Plutonium (²⁴¹ Pu)	Water	1	HDPE	≤6 months	HNO ₃ to pH < 2
Strontium (⁹⁰ Sr)	Water	0.5–1	HDPE	≤6 months	HNO ₃ to pH < 2
Technetium (⁹⁹ Tc)	Water	0.5–2	HDPE	≤6 months	HNO ₃ to pH < 2
Tritium (³ H)	Water	0.1–0.5	HDPE/Glass ^e	≤6 months	None

a. Volumes vary depending on the requested analysis and the laboratory performing the analysis. Exact volumes required will be specified to project personnel following final determination of the analytical services provider. Any additional volume to allow for analysis of duplicates required by the analytical method will also be specified by the laboratory before sampling.

b. HDPE = High-density polyethylene.

c. The holding time requirement of six months is described in 40 CFR 136 (2003) (EPA guidelines for analysis of pollutants) and is applied as a general guideline. For analysis of volatile radionuclides not listed above or radionuclides with short half-lives (e.g., ¹³¹I), the holding times will be adjusted accordingly and disseminated to the laboratory via a project-specific statement of work.

d. Collecting samples for ¹²⁹I in HDPE containers is permissible/acceptable; however, the holding time requirement is 28 days (instead of 6 months).

e. Samples containing high levels of tritium (³H) must be collected in glass containers. High-level ³H is defined as concentrations that exceed 10⁴ pCi/L.

Table 7. Summary of sample collection, holding time, and preservation requirements for nonradiological analyses.

Analysis ^a	Sample Medium	Volume ^b	Container Type ^c	Holding Time	Preservative
Anions	Residual Solid	250 mL	Wide-mouth glass jar	28 days ^d	4°C ^d
Anions	Water	500 mL	HDPE bottle	Analyze within 48 hours for NO ₃ and PO ₄ . All others within 28 days ^d	4°C ^d
Methanol	Water	1 × 40 mL ^e	40-mL glass vial, Teflon-lined cap	Analyze within 14 days ^f	4°C (add H ₂ SO ₄ , HCl, or NaHSO ₄ to pH <2, as necessary) ^f
PCBs	Water	1,000 mL ^e	Amber-colored glass jugs, Teflon-lined cap	Extract within 7 days, analyze extracts within 40 days of extraction ^f	4°C ^f
pH	Water	60 mL	HDPE	Analyze within 24 hours	None
TCLP Metals	Residual Solid	500 mL	Wide-mouth glass jar	For metals, except Hg: (a) complete TCLP extraction within 6 months; and (b) complete determinative analysis (DA) within 6 months of TCLP extraction. For Hg: (a) complete TCLP extraction within 28 days; and (b) complete DA within 28 days of TCLP extraction ^f	4°C ^f
Total Metals	Residual Solid	125 mL	Wide-mouth glass jar	Analyze within 6 months, except Hg analyze within 28 days ^f	4°C ^f
Total Metals	Water	1,000 mL	HDPE bottle	Analyze within 6 months, except Hg analyze within 28 days ^f	HNO ₃ to pH <2 ^f
Total SVOCs	Water	1,000 mL ^e	Amber-colored glass jugs, Teflon-lined cap	Extract within 7 days, analyze extracts within 40 days of extraction ^f	4°C ^f
Total SVOCs and PCBs	Residual Solid	250 mL	Wide-mouth glass jar, Teflon-lined cap	Extract within 14 days, analyze extracts within 40 days of extraction ^f	4°C ^f
Total VOCs	Water	2 × 40 mL ^e	40-mL glass vial, Teflon-lined cap ^e	Analyze within 14 days ^f	4°C (add H ₂ SO ₄ , HCl, or NaHSO ₄ to pH <2, as necessary) ^f
Total VOCs	Residual Solid	125 mL	Wide-mouth glass jar, Teflon-lined cap	Analyze within 14 days ^f	4°C ^f

a. PCB = Polychlorinated biphenyl

SVOC = Semivolatile organic compound

VOC = Volatile organic compound.

b. Volumes may vary depending on the requested analysis and the laboratory performing the analysis. Exact volumes required will be specified to project personnel following final determination of the analytical services provider. Any additional volume to allow for analysis of quality control samples required by the analytical method will also be specified by the laboratory before sampling. The minimum volume required to meet the required detection limits will be collected for each analysis to ensure personnel radiation exposure is maintained as low as reasonably achievable.

c. It is highly recommended that a certificate of cleanliness be obtained for all lots of sample containers used.

d. Source: *Methods for Chemical Analysis of Water and Wastes* (EPA 1983).

e. Once each 20 samples or 14 days, whichever comes first, three times the normal sample volume is required (e.g., 3,000 mL instead of 1,000 mL, 6 H 40 mL instead of 2 H 40 mL, etc.).

f. Source: SW-846, Chapter 2 (EPA 1998).

For solid sample collection at one location, the individual sample bottles will be decanted or filtered in the laboratory and the solids will be combined for a composite sample of the solids from that location. If hazardous constituent analyses are required, this composite sample will be analyzed as a single sample representing each location where greater than 15% solids are present. The relative volumes of the liquid and solid phases will be estimated to apply the TCLP calculations for total analyte concentration, as discussed previously. If only radionuclide analyses are required (i.e., the volume of solid is <15%), the individual aliquots from each location in the tank will be composited (i.e., a total of five composited aliquots) in a jar of appropriate geometry for the gamma spectroscopy and radiochemical analyses required. Gamma spectroscopy, which is a nondestructive analytical technique, will be conducted first, after which the radionuclide analyses (in which the sample is consumed) will be performed.

If the simple sampler is used, more than one simple sampler volume will be needed to fill all the bottles required to complete the specified sample analyses. The simple sampler will be lowered into the tank several times at each location until adequate volume is collected. This process will be repeated at all five sample collection locations in the tanks. The sample apportionment process will assume a relatively homogeneous liquid sample material is being retrieved from the tank. Therefore, no sample compositing of liquids will take place during the sample apportionment process unless it is required to ensure adequate sample volume for analysis. The individual sample bottles will be filled in the RAL directly from the 1-L sample collection vessels.

If the submersible pump is used, it will only have to be lowered through each sample access riser one time to obtain the volume required to complete the analyses listed in Tables 6 and 7. The submersible pump will be activated repeatedly until enough 1-L bottles are filled to obtain the volume required. This process will be repeated at all five sample collection locations in the tanks. The sample apportionment process will assume a relatively homogeneous liquid sample material is being retrieved from the tank. Therefore, no sample compositing of liquids will take place during the sample apportionment process unless it is required to ensure adequate sample volume for analysis. The individual sample bottles will be filled in the RAL directly from the 1-L sample collection bottles.

Post-decontamination rinsate from the pipe encasements will be allowed to drain into the WM-181 vault floor and into the WM-181 vault sump and a sample of the combined rinsate solution collected from the sump.

Prior to flushing, the cooling coils in Tanks WM-103 through WM-106 will be pressure tested. If pressure testing of the cooling coils confirms their integrity, the coils will be flushed and samples collected using the existing line valves present in CPP-634. If pressure testing determines that the integrity of the coils is uncertain, flushing will not be performed. Instead, a 2-in. section of the coils will be removed, rinsed, and the rinsate sampled.

- Five samples will be collected from the cooling coils in Tanks WM-103 through WM-106. The sample locations for the cooling coils were chosen using a random numbers table to ensure that random sampling statistics can be applied to the data collected. The sample locations are cooling coils WRA 10, WRA 14, WRA 17, WRA 29, and WRA 34. (The cooling coil number refers to the number assigned to the cooling coil return lines.)

5.1.3 Sample Preservation

Sample preservation ensures that target analytes do not escape from field samples or become chemically attached to sample containers before analysis. Typical sample preservation activities include the addition of acids to ensure that metals and radionuclides remain in solution and to inhibit biological

activity that could affect organic constituents. To prevent volatile and semivolatile materials from escaping sample media, samples are cooled to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

Radioactivity levels in the samples require delivery to the RAL before preservation. It is expected that the turnaround of samples from collection to delivery at the RAL will be a very short period of time (a matter of minutes). On receipt at the laboratory, the materials will be split and placed in refrigerators or coolers. Ice or blue ice used to cool samples may become an investigation-derived waste after contact with WM-103 through WM-106 and WM-181 radiation fields and other hazardous constituents. If so, it must be managed thereafter as a waste form. Therefore, because the samples will be transported to the RAL before apportioning, ice will not be used to cool the samples between collection and delivery to prevent creation of additional waste.

During sample apportioning, the sampling team member or RAL analyst will first transfer sufficient liquid for VOC determination to two 40-mL glass vials for each sample and samples collected for methanol to one separate 40-mL glass vial (as defined in Chapter 2 of SW-846 and listed in Table 7). For the liquid samples to be used for VOC determination, the sampling team member or analyst will ensure that no headspace remains in the sample vials after the caps are placed on them, and the team will ensure the liquid is agitated as little as possible during transport. Headspace is checked by inverting the bottle and lightly tapping on the lid to ensure no bubbles are visible in the container. The samples will then be labeled and cooled.

The sampling team member or RAL analyst shall inspect the individual sample matrices generated during the apportionment process to determine if each sample phase contains sufficient material to perform the requested analyses. The individual matrices must be placed in glass or high-density polyethylene (HDPE) containers and preserved as described in Tables 6 and 7 before transport to the laboratory performing the analyses.

5.1.4 Field Radiological Control Screening

Because of the potential intensity of radiation fields at Tanks WM-103 through WM-106 and WM-181, all sampling and analysis activities will comply with INEEL MCPs and RAL standard operating procedures for those samples shipped to the RAL. The radiological controls and personnel monitoring requirements established for the simple sampler, submersible pump, or other appropriate sampling equipment operation, and the subsequent sample transfer, are based on radiation exposure rates calculated using process data obtained during the operation of Tanks WM-103 through WM-106 and WM-181. These exposure rates will be used to implement action levels that will help ensure that all work activities and personnel exposures to direct radiation are maintained ALARA (PRD-183, 2000).

In addition to monitoring for personnel exposure, the simple sampler and submersible pump also will be directly monitored at several points throughout the collection process to make decisions relative to how the samples will be delivered to the RAL for sample apportionment (i.e., shielding requirements). Two separate means of monitoring the radiation field associated with the samples will be used. First, telemetry dosimeters with remote read-out capabilities will be placed in locations that will be readily exposed to the radiation field during sample extraction. Second, following an evaluation by the IH, the RCT will use hand-held instrumentation to screen beta and gamma radiation in the sample collection port.

These direct radiation screenings will be used to determine whether the sample is suitable for direct handling and apportioning activities or if it must be shielded for transport and delivery to the RAL. All activities relating to the post-decontamination characterization of the WM-103 through WM-106 and WM-181 tank systems will be done in accordance with requirements of PRD-183, "INEEL Radiological Control Manual" (2000).

5.1.5 Sample Containers

It is possible that all samples from the tanks and WM-181 vault sump will be collected in the simple sampler chamber or by using the submersible pump. The submersible pump uses vacuum pressure to draw materials into an appropriate receiving vessel. The simple sampler uses vacuum pressure to draw materials into a stainless steel tube. If the simple sampler is used, appropriate shielding will be used after the sampler is retrieved from the riser. Approximately 200 mL of solid/liquid mixture will likely be obtained during each simple sampler trip. The volumes of samples collected using the submersible pump is limited only by the size of the available sample containment vessel. Using these types of samplers introduces the potential for data usability limitations for organic analyses. Specifically, the use of a vacuum to draw a sample for VOAs could result in an unquantifiable loss of analytes of interest. However, the loss of analytes attributable to the use of the vacuum collection system is likely negligible compared to the loss resulting from the highly acidic and elevated temperature conditions of the waste in the tank.

The use of an HDPE bottle could introduce levels of phthalate esters such that dilution of the sample would be required during analysis for semivolatile organic compounds (SVOCs). Therefore, samples collected for SVOC analyses will not come in contact with HDPE containers. A Teflon or glass container will be used to obtain samples for SVOC analysis.

The samples will be transported to the RAL by field personnel. The RAL analyst will transfer sufficient liquid for VOC determination to 40-mL glass vial(s) for the methanol determination by EPA Method 8015B analysis and separate 40-mL glass vials to determine the other required VOCs by EPA Method 8260B, and will acidify the samples (as defined in Chapter 2 of SW-846 and listed in Table 7). If the samples are to be transported to another laboratory for analysis, the samples will be placed in a shipping container and cooled to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. The RAL technical staff will inspect the individual sample matrices generated during the phase separation process to determine if each sample phase contains sufficient material to perform the requested analysis. Samples determined to contain insufficient material to perform the requested analysis must be composited with materials of the same matrix from other samples collected from the same location. The need for additional samples will be determined by the laboratory and the PM. The individual matrices must be placed in the appropriate analysis-specific glass or HDPE containers and preserved as described in Tables 6 and 7 before transport to the analytical laboratory performing the analyses.

5.1.6 Sample Transport

Upon completion of sample retrieval from the WM-103 through WM-106 and WM-181 tank heels and the WM-181 vault sump, appropriate precautions will be used to seal the sample collection container that houses the liquid and solid phases of material. The sealed container will then be approached and, if within safety limits, scanned by an RCT using hand-held radiation survey equipment. If the activity of the container is too high to allow the RCT to approach it, the container will be placed inside a shielded secondary transport vessel, which will be sealed. The sample and the containment vessel will be transported to a vehicle by hand or by using a handcart equipped with a lock-down strap, and then be transported to the RAL for turnover to RAL personnel.

Because the samples from the tank heels, and/or vault sump will be transported to the RAL for sample apportionment, field and trip blanks will not be introduced before transport of the sample to the RAL. The field and trip blanks would have little use because they would be handled differently than the sample. Because the field and trip blanks would not enter the transport vessel or the hot cell, the analysis of these blanks would not represent contamination introduced during transport of these samples. If

aliquots of the samples will be shipped offsite for VOC analyses, trip blanks will be introduced in sample shipping containers before shipment.

Trip blanks are organic-free water in a 40-mL vial, sent from the laboratory that will be performing the analyses, that accompanies VOC water samples during the sample collection and shipment processes. An alternative source of trip-blank water is from the INEEL Analytical Laboratories Department where reagent water that has been heated and pre-purged with an inert gas is available for use as trip-blank media. Trip blanks evaluate cross-contamination during sample handling, shipment, and storage.

Field blanks are analyte-free water poured into a sample container at the sample collection site to check cross-contamination during sample collection and shipment. Field blanks are often not collected during waste sampling activities because the very low level of cross contamination detectable using field blanks would not affect a decision concerning data obtained from measurements on a concentrated waste.

Aliquots of the samples to be shipped for offsite analyses will require gamma-screen. These samples will be shipped according to all applicable U.S. Department of Transportation requirements.

5.1.7 Waste Management

Wastes generated as a result of the post-decontamination characterization of the WM-103 through WM-106 and WM-181 tank system components will include laboratory wastes and waste generated from decontamination of the sample sampler sample chamber and/or submersible pump. Field wastes in the form of paper towels and other wastes associated with the sample apportionment activities will be generated as a result of sample collection. The INEEL Waste Generator Services will ensure that disposition of non-sample waste material is in compliance with the HWMA/RCRA closure plan and that applicable paperwork is completed. All samples and analysis wastes disposed of by the INEEL Analytical Chemistry Laboratory will be disposed of through normal routes and in accordance with INEEL MCP-3480, "Environmental Instructions for Facilities, Processes, Materials and Equipment" (2002). The Waste Generator Services waste technical specialist will ensure compliance with the applicable HWMA/RCRA requirements; ISD-9, "INTEC RCRA Interim Status Document for the Process Equipment Waste Evaporator System, Section B – Waste Analysis Plan" (2002); MCP-62, "Waste Generator Services Low-Level Waste Management" (2003); and MCP-70, "Mixed Low-Level Waste Management" (2003).

6. SAMPLING PROCEDURES

Because of the limited access and nature of the sample material, post-decontamination characterization samples will be collected from tank heels and the WM-181 vault sump using a simple sampler, submersible pump, or other appropriate sampling equipment. Samples of rinse solutions from the cooling coils in Tanks WM-103 through WM-106 will be collected using existing line valves present in CPP-634, or by removing sections of the cooling coils, as explained earlier. Other possible sampling approaches include:

- Collecting decontamination rinsate or heel samples at the discharge of the steam jet pumps used for heel reduction
- Collecting liquid samples through risers with bailers or other grab sampling techniques.

If an alternative sampling approach is chosen, the specific procedures relevant to the chosen approach will be incorporated in a revision to this SAP. This document assumes the use of the simple sampler, submersible pump, or other remote sampling equipment to collect samples from the tanks at the locations specified in Section 5.1.2, and the use of existing line valves present in CPP-634 for sampling the cooling coils or removal of cooling coil sections prior to sampling. No waste transfer lines will be sampled for Tanks WM-103 through WM-106 and WM-181. The sampling procedures for the waste transfer lines discussed in the *Sampling and Analysis Plan for the Post-Decontamination Characterization of the Process Waste Lines from INTEC Tank Farm Facility Tanks WM-182 and WM-183* (INEEL 2001) is assumed to represent all waste transfer lines in the TFF.

6.1 Simple Sampler

The simple sampler consists of a sample collection chamber attached to a line that can be lowered into the tank risers. The sample collection chamber is narrow enough that it can fit through the 2-in. pipe that is part of two riser assemblies accessible in each of the three tanks. The lines that lower the sampler to the floor of the tank apply a vacuum to the chamber, using a bimba tube, allowing tank contents to enter the sampler nose and fill the chamber. Tank WM-182 sampling activities and cold tests of the simple sampler showed it could collect solids and liquids in very close proportion to the known amounts of each of these phases in the test solutions.

The sample chamber capacity is roughly 250 or 500 mL. The sample chamber will be filled to the maximum extent possible during each simple sampler trip. The sample chamber will be surveyed by an RCT to ensure that exposure for sampling team members and decontamination personnel are maintained ALARA.

To ensure the integrity of samples collected from the tank access risers, the simple sampler will be cleaned before first use and between discrete sampling events. Adequate cleaning typically comprises triple rinsing the sampling equipment with de-ionized water.

6.2 Submersible Pump

The submersible pump consists of a narrow diameter polymer pump attached to Teflon tubing that can be lowered into tank risers for sample collection. The pump uses negative pressure to pull sample material into a sample vessel located above the tank contents. The submersible pump will be used to collect volumetric samples for liquids overlaying residual solids in the tank heel and to collect solid-liquid mixtures from the bottom of the tank heel at the liquid-solid interface. The sample bottles will be

surveyed by an RCT to ensure that exposure for sampling team members and decontamination personnel are maintained ALARA.

To ensure the integrity of samples collected from the tank access risers, the designated submersible pump and associated tubing will be flushed between discrete sampling events. To accomplish this, the submersible pump will purge a sufficient volume of sampling liquid before the sample collection. The surfaces of the pump and associated tubing that contact the post-decontamination rinsate during sample collection will be primed before sample collection using the agitated tank contents.

6.3 Sample Collection Procedures

To ensure that all samples are collected in a comparable way from sampling effort to sampling effort, the following operating procedures are used to deploy and sample with the submersible pump and/or simple sampler:

- For the collection of samples from tank sumps, TPR-7094, “Take Tank Farm Sump Samples” (2003), will be used to prepare the sampling systems for deployment, operate the appropriate sampling system, transport samples, and conduct other supporting activities
- For the collection of samples from residual tank heels, TPR-7095, “Take Tank Farm Vessel Samples (Post Decon)” (2003), will be used to prepare the sampling systems for deployment, operate the appropriate sampling system, transport samples, and conduct other supporting activities.

The radiological controls and personnel monitoring requirements established for direct simple sampling and submersible pump operations and the subsequent sample transfer are based on calculated radiation exposure rates. The calculated exposure rates were based on historical sample-analysis records for comparable Tanks WM-182 and WM-183. As a result, the operating procedures and associated radiological work permit tasks issued for the work will implement and/or specify action levels to ensure all work activities and personnel exposure to direct radiation are maintained ALARA. Any decision to resample will be made with authorization from the appropriate responsible facility, operational, and program personnel located at the job site. If the simple sampler is used, radiation measurements will be taken after the simple sampler is retrieved from the riser. If the submersible pump is used, radiation measurements will be taken while the sample is accumulating in the sample vessel. Radiation measurements will be taken using instruments under control of the INTEC RCTs.

The direct radiation readings will be recorded in the field logbook. Field manipulation of the sample will be based on the total activity of the sampled liquid as defined in the *Idaho Nuclear Technology and Engineering Center Safety Analysis Report* (INEEL 1999). The RCT’s evaluation of the sample’s radiation levels ensures the sample will comply with the Safety Analysis Report and the “INEEL Radiological Control Manual” (PRD-183, 2000) requirements.

Because more volume is needed than can be obtained in one attempt using the simple sampler, the retrieved sample volume will be transferred into 1-L bottles and another trip will be made to obtain more sample material. Additional sample volume can be obtained by reusing the original sample chamber. No decontamination will be required between trips to the tank as long as each subsequent trip is for the purpose of obtaining additional volume for the same sample. A new or fully-cleaned sample chamber will be used at every new sample location.

If pressure testing of the cooling coils in Tanks WM-103 through WM-106 confirms their integrity, the coils will be flushed and samples of rinsate collected directly into the appropriate bottles using

existing line valves present in CPP-634. If pressure testing determines the integrity of the coils are uncertain, 2-in. sections of pipe will be removed. The pipe sections will be filled with deionized water and allowed to equilibrate for 30 minutes. Samples of the rinsate will then be collected directly into the appropriate sample bottles. The major contaminant of the cooling coils is chromium, which was used as a corrosion inhibitor. Five cooling coil samples will be collected and analyzed for chromium by the methods specified in Table 5.

Pipe encasements will be rinsed to decontaminate the interior surfaces. The rinsate solution will drain to the WM-181 vault sump, and a sample representing the composite of the pipe encasements and the vault floor rinsate will be collected from the vault sump. The sample collected from the vault sump will be analyzed for total metals, VOCs, SVOCs, polychlorinated biphenyls (PCBs), methanol, radionuclides, and anions by the methods specified in Table 5.

Table 8 provides a summation of the samples that are anticipated to be collected during the sampling efforts and includes the number of anticipated samples, anticipated collection dates, and the analytes anticipated to be requested for each sample. Because the samples must be transported to the RAL for sample apportionment, field and trip blanks will not be collected (as stated in Section 5.1.6, “Sample Transport”).

Table 8. Anticipated sample collection from each of the WM-103 through WM-106 and WM-181 tank systems.

Analysis	Estimated Number of Samples from Each Tank ^a	WM-181 Vault Sump ^b	Tanks WM-103 through -106 Cooling System	Matrix ^c	Analytes of Interest	Dates of Collection
Anions	5	1		Liquid	Cl, F, PO ₄ , NO ₃ , and SO ₄	TBD ^d
Methanol	5	1		Liquid	EPA Method 8015B using a direct injection technique for methanol	TBD
PCBs	5	1		Solid	EPA Method 8082 (see Table 10)	TBD
PCBs	5	1		Liquid	EPA Method 8082 (see Table 11)	TBD
pH	5	1	5	Liquid	pH	TBD
Radionuclides	5	1	5 (γ only)	Liquid	²⁴¹ Am, ¹⁴ C, ⁶⁰ Co, ¹³⁴ , ¹³⁷ Cs, ³ H, ¹²⁹ I, ²³⁷ Np, ⁶³ Ni, ⁹⁰ Sr, ⁹⁹ Tc, ⁹⁴ Nb, ¹⁵⁵ , ¹⁵⁴ Eu, ²⁴⁴ Cm, ²³⁸ , ^{239/240} , ²⁴¹ Pu, and ²³⁴ , ²³⁵ , ²³⁶ , ²³⁸ U	TBD
Radionuclides ^e	5	1		Solid	²⁴¹ Am, ¹⁴ C, ⁶⁰ Co, ¹³⁴ , ¹³⁷ Cs, ³ H, ¹²⁹ I, ²³⁷ Np, ⁶³ Ni, ⁹⁰ Sr, ⁹⁹ Tc, ⁹⁴ Nb, ¹⁵⁵ , ¹⁵⁴ Eu, ²⁴⁴ Cm, ²³⁸ , ^{239/240} , ²⁴¹ Pu, and ²³⁴ , ²³⁵ , ²³⁶ , ²³⁸ U	TBD
Semivolatile Organic Compounds	5	1		Solid	EPA Method 8270C (see Table 10)	TBD
Semivolatile Organics	5	1		Liquid	EPA Method 8270C (see Table 11)	TBD
Total Metals ^f	5	1	5	Liquid Solid	Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Tl, V, and Zn	TBD
Toxicity Characteristic Leaching Procedure Metals	5			Solid	Ag, As, Ba, Cd, Cr, Hg, Pb, and Se	TBD
Volatile Organic Compounds	5	1		Solid	EPA Method 8260B (see Table 10)	TBD
Volatile Organics	5	1		Liquid	EPA Method 8260B (see Table 11)	TBD

a. The number of samples does not include trip blanks and field blanks. Trip blanks will be collected for VOC analyses only, one for each cooler shipped with VOC samples in them. Field blanks will not be collected.

b. One sample collected from Tank WM-181 vault sump following final rinse of the vault floor and one additional sample from each selected DVB sump. (A minimum of two discrete DVBs must be sampled during this characterization effort.)

c. Solid material will only be analyzed if present at greater than 15% of the total sample volume retrieved.

d. TBD = To be determined.

e. This includes the radionuclides that will contribute the greatest dose from the list of key radionuclides as described in DOE Manual 435.1-1, Chapter II (2001).

f. For samples collected from the cooling coils, chromium is the only analyte of interest.

7. ANALYTICAL METHODS

To ensure that data of acceptable quality are obtained from the post-decontamination characterization of the WM-103 through WM-106 and WM-181 tank system components, standard EPA laboratory methods or technically appropriate methods for radioanalytical determinations will be used to obtain project laboratory data. Analytical measurements and the reporting protocols that will be used to determine inorganic, organic, and radiochemical constituents are outlined in Table 9.

The TCLP metals sample preparation and analysis of solids retrieved from the tanks will be performed on the WM-103 through WM-106 and WM-181 samples using the methods listed in Table 9. The TCLP method specifies that samples with less than 0.5% solids do not require extraction. If less than 15% of the total sample mass retrieved is solid material, no hazardous constituent analyses will be conducted on solid samples. Radionuclide analyses will be required for all solids and liquids collected. The TCLP typically requires a 100-g sample. The IDEQ has authorized the INEEL to allow laboratories to use smaller amounts as a sample when ALARA concerns exist. The laboratory SOW will specify that if a smaller sample mass will be used, the PM must be contacted to authorize this action. Project personnel will consult with persons cognizant of laboratory methods to ensure the impacts to method detection limits caused by using a smaller sample volume will not adversely impact the data use relative to the project DQOs.

Determinations for total inorganic and organic constituents will be performed by the methods presented in *Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods* (EPA 1998) and listed in Table 9. Radiological determinations will be performed according to approved methods and ER-SOW-394 (2002) requirements.

Tables 10 and 11 summarize the method-specific requirements that will be followed by the analytical laboratory to the extent possible given the sample restrictions. Any deviations from this information will be fully documented, and the PQAO and PM will be informed of deviations.

7.1 Analytical Laboratory

The laboratory chosen for conducting the analyses will have the appropriate level of qualified personnel, appropriate instrumentation, an approved QA plan, approved analytical methods, and appropriate internal standard operating procedures to perform the required analyses. The selected laboratory will be approved for use as documented by their inclusion on the INEEL-approved suppliers list. The QA plans and standard operating procedures for the laboratory (or laboratories) selected for performing the required analyses will be available for review by project personnel.

Table 9. Analytical method source documents and method descriptions.

Inorganic and Organic Determinations	
Method Number	Title
1311 ^a	Toxicity Characteristic Leaching Procedure
3010A ^a	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by Flame Atomic Absorption Spectrometry or Inductively Coupled Plasma Spectroscopy
3050B ^a	Acid Digestion of Sediments, Sludges, and Soils
3520C ^a	Continuous Liquid-Liquid Extraction
3540C ^a	Soxhlet Extraction
5030B ^a	Purge-and-Trap for Aqueous Samples
5031 ^b	Volatile, Nonpurgeable, Water-Soluble Compounds by Azeotropic Distillation
5035 ^a	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
6010B ^a	Inductively Coupled Plasma-Atomic Emission Spectroscopy
7060A ^a	Arsenic (Atomic Absorption, Furnace Technique)
7470A ^a	Mercury in Liquid Waste (Manual Cold-Vapor Technique)
7471A ^a	Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)
7740 ^a	Selenium (Atomic Absorption, Furnace Technique)
8015B ^b	Nonhalogenated Organics Using Gas Chromatography/Flame Ionization Detector
8082 ^a	Polychlorinated Biphenyls by Gas Chromatography
8108 ^c	Total Dissolution Sample Preparation
8260B ^a	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry
8270C ^a	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry
9040B ^a	pH Electrometric Measurement
9045C	Soil and Waste pH
9056 ^a	Determination of Inorganic Anions by Ion Chromatography
Radiochemical Determinations	
Method Number	Description
ER-SOW-394 ^d	Determination of Radionuclides

a. Source: EPA 1998.

b. The laboratory will perform Method 5031 before the analysis of samples using Method 8015B.

c. Source: *INEEL Analytical Chemistry Methods Manual* (INEEL 2003).

d. Source: ER-SOW-394 (2002).

Table 10. Sample preparation, analytical methods, and recommended detection limits—solids.

Analysis TCLP Extraction	Recommended Detection Limit Not Applicable	Preparation Method SW-846 1311	Analysis Method Not Applicable
TCLP Metals Analysis	(mg/L)		
Arsenic	40	SW-846 3010A	SW-846 6010B
Barium	40	SW-846 3010A	SW-846 6010B
Cadmium	1	SW-846 3010A	SW-846 6010B
Chromium	2	SW-846 3010A	SW-846 6010B
Lead	0.6	SW-846 3010A	SW-846 6010B
Mercury	0.04	SW-846 7470A	SW-846 7471A
Selenium	1	SW-846 3010A	SW-846 6010B
Silver	2	SW-846 3010A	SW-846 6010B
Total Metals	(mg/kg)		
Aluminum	60	SW-846 3050B	SW-846 6010B
Antimony	40	SW-846 3050B	SW-846 6010B
Arsenic	2	SW-846 3050B	SW-846 7060A
Barium	2	SW-846 3050B	SW-846 6010B
Beryllium	0.4	SW-846 3050B	SW-846 6010B
Cadmium	0.4	SW-846 3050B	SW-846 6010B
Calcium	1.4	SW-846 3050B	SW-846 6010B
Chromium	1	SW-846 3050B	SW-846 6010B
Cobalt	1	SW-846 3050B	SW-846 6010B
Copper	0.8	SW-846 3050B	SW-846 6010B
Iron	8	SW-846 3050B	SW-846 6010B
Lead	56	SW-846 3050B	SW-846 6010B
Manganese	1.8	SW-846 3050B	SW-846 6010B
Mercury	0.04	SW-846 7471A	SW-846 7471A
Nickel	20	SW-846 3050B	SW-846 6010B
Selenium	34	SW-846 3050B	SW-846 7740
Silver	10	SW-846 3050B	SW-846 6010B
Thallium	60	SW-846 3050B	SW-846 6010B
Vanadium	10	SW-846 3050B	SW-846 6010B
Zinc	2	SW-846 3050B	SW-846 6010B
Radionuclides	(pCi/g)		
²⁴¹ Am	0.2	ER-SOW-394	ER-SOW-394
¹⁴ C	3	ER-SOW-394	ER-SOW-394
²⁴⁴ Cm	0.05	ER-SOW-394	ER-SOW-394
³ H	20	ER-SOW-394	ER-SOW-394
¹²⁹ I	1	ER-SOW-394	ER-SOW-394
⁶³ Ni	5	ER-SOW-394	ER-SOW-394
²³⁷ Np	0.05	ER-SOW-394	ER-SOW-394
^{238, 239/240} Pu	0.05	ER-SOW-394	ER-SOW-394
²⁴¹ Pu	1	ER-SOW-394	ER-SOW-394
⁹⁰ Sr	0.5	ER-SOW-394	ER-SOW-394
⁹⁹ Tc	1	ER-SOW-394	ER-SOW-394
U isotopic	0.05	ER-SOW-394	ER-SOW-394
Gamma-emitting radionuclides: ⁶⁰ Co, ^{134, 137} Cs, ⁹⁴ Nb, ^{154, 155} Eu	0.1 ^a	ER-SOW-394	ER-SOW-394

Table 10. (continued).

Analysis	Recommended Detection Limit	Preparation Method	Analysis Method
TCLP Extraction	Not Applicable	SW-846 1311	Not Applicable
Organic Constituents			
Volatiles	(mg/kg)		
2-Butanone	2	SW-846 5035	SW-846 8260B
1,1-Dichloroethene	2	SW-846 5035	SW-846 8260B
1,2-Dibromoethane	2	SW-846 5035	SW-846 8260B
1,2-Dibromo-3-chloropropane	2	SW-846 5035	SW-846 8260B
1,2-Dichlorobenzene	2	SW-846 5035	SW-846 8260B
1,3-Dichlorobenzene	2	SW-846 5035	SW-846 8260B
1,4-Dichlorobenzene	2	SW-846 5035	SW-846 8260B
1,2-Dichloropropane	2	SW-846 5035	SW-846 8260B
2-Hexanone	2	SW-846 5035	SW-846 8260B
4-Methyl-2-pentanone	2	SW-846 5035	SW-846 8260B
1,1,2,2-Tetrachloroethane	2	SW-846 5035	SW-846 8260B
1,2,4-Trichlorobenzene	2	SW-846 5035	SW-846 8260B
1,1,1-Trichloroethane	2	SW-846 5035	SW-846 8260B
1,1,2-Trichloroethane	2	SW-846 5035	SW-846 8260B
1,1,2-Trichloro-1,2,2-trifluoroethane	2	SW-846 5035	SW-846 8260B
Acetone	2	SW-846 5035	SW-846 8260B
Benzene	2	SW-846 5035	SW-846 8260B
Bromodichloromethane	2	SW-846 5035	SW-846 8260B
Bromoform	2	SW-846 5035	SW-846 8260B
Bromomethane	2	SW-846 5035	SW-846 8260B
Carbon Disulfide	2	SW-846 5035	SW-846 8260B
Carbon Tetrachloride	2	SW-846 5035	SW-846 8260B
Chlorobenzene	2	SW-846 5035	SW-846 8260B
Chloroethane	2	SW-846 5035	SW-846 8260B
Chloroform	2	SW-846 5035	SW-846 8260B
Chloromethane	2	SW-846 5035	SW-846 8260B
cis-1,2-Dichloroethene	2	SW-846 5035	SW-846 8260B
cis-1,3-Dichloropropene	2	SW-846 5035	SW-846 8260B
Cyclohexane	2	SW-846 5035	SW-846 8260B
Cyclohexanone	1.4	SW-846 5035	SW-846 8260B
Dibromochloromethane	2	SW-846 5035	SW-846 8260B
Dichlorodifluoromethane	2	SW-846 5035	SW-846 8260B
Ethyl acetate	5	SW-846 5035	SW-846 8260B
Ethylbenzene	2	SW-846 5035	SW-846 8260B
Isopropylbenzene	2	SW-846 5035	SW-846 8260B
Methanol	5	SW-846 8015B	SW-846 8015B
Methyl Acetate	2	SW-846 5035	SW-846 8260B
Methylcyclohexane	2	SW-846 5035	SW-846 8260B
Methylene Chloride	2	SW-846 5035	SW-846 8260B
Styrene	2	SW-846 5035	SW-846 8260B
Tetrachloroethene	2	SW-846 5035	SW-846 8260B
Toluene	2	SW-846 5035	SW-846 8260B
Trans-1,2-Dichloroethene	2	SW-846 5035	SW-846 8260B
Trans-1,3-Dichloropropene	2	SW-846 5035	SW-846 8260B
Trichloroethene	2	SW-846 5035	SW-846 8260B
Trichlorofluoromethane	2	SW-846 5035	SW-846 8260B
Vinyl Chloride	2	SW-846 5035	SW-846 8260B

Table 10. (continued).

Analysis	Recommended Detection Limit	Preparation Method	Analysis Method
TCLP Extraction	Not Applicable	SW-846 1311	Not Applicable
Xylenes (Total)	2	SW-846 5035	SW-846 8260B
Semivolatiles	(mg/kg)		
1,1'-Biphenyl	2	SW-846 3540C	SW-846 8270C
4-Bromophenyl-phenylether	2	SW-846 3540C	SW-846 8270C
4-Chloroaniline	2	SW-846 3540C	SW-846 8270C
2-Chloronaphthalene	2	SW-846 3540C	SW-846 8270C
2-Chlorophenol	2	SW-846 3540C	SW-846 8270C
4-Chlorophenyl-phenylether	2	SW-846 3540C	SW-846 8270C
4-Chloro-3-methylphenol	2	SW-846 3540C	SW-846 8270C
3,3'-Dichlorobenzidine	2	SW-846 3540C	SW-846 8270C
2,4-Dichlorophenol	2	SW-846 3540C	SW-846 8270C
2,4-Dimethylphenol	2	SW-846 3540C	SW-846 8270C
2,4-Dinitrophenol	5	SW-846 3540C	SW-846 8270C
2,4-Dinitrotoluene	2	SW-846 3540C	SW-846 8270C
2,6-Dinitrotoluene	2	SW-846 3540C	SW-846 8270C
4,6-Dinitro-2-methylphenol	5	SW-846 3540C	SW-846 8270C
2-Methylnaphthalene	2	SW-846 3540C	SW-846 8270C
2-Methylphenol	2	SW-846 3540C	SW-846 8270C
4-Methylphenol	2	SW-846 3540C	SW-846 8270C
2-Nitroaniline	5	SW-846 3540C	SW-846 8270C
3-Nitroaniline	5	SW-846 3540C	SW-846 8270C
4-Nitroaniline	5	SW-846 3540C	SW-846 8270C
2-Nitrophenol	2	SW-846 3540C	SW-846 8270C
4-Nitrophenol	5	SW-846 3540C	SW-846 8270C
2,2'-Oxybis (1-Chloropropane)	2	SW-846 3540C	SW-846 8270C
2,4,5-Trichlorophenol	5	SW-846 3540C	SW-846 8270C
2,4,6-Trichlorophenol	2	SW-846 3540C	SW-846 8270C
Acenaphthene	2	SW-846 3540C	SW-846 8270C
Acenaphthylene	2	SW-846 3540C	SW-846 8270C
Acetophenone	2	SW-846 3540C	SW-846 8270C
Anthracene	2	SW-846 3540C	SW-846 8270C
Atrazine	2	SW-846 3540C	SW-846 8270C
Benzaldehyde	2	SW-846 3540C	SW-846 8270C
Benzo(a)anthracene	2	SW-846 3540C	SW-846 8270C
Benzo(a)pyrene	2	SW-846 3540C	SW-846 8270C
Benzo(b)fluoranthene	2	SW-846 3540C	SW-846 8270C
Benzo(g,h,i)perylene	2	SW-846 3540C	SW-846 8270C
Benzo(k)fluoranthene	2	SW-846 3540C	SW-846 8270C
bis-(2-Chloroethoxy) methane	2	SW-846 3540C	SW-846 8270C
bis-(2-Chloroethyl)ether	2	SW-846 3540C	SW-846 8270C
bis-(2-Ethylhexyl)phthalate	2	SW-846 3540C	SW-846 8270C
Butylbenzylphthalate	2	SW-846 3540C	SW-846 8270C
Caprolactam	2	SW-846 3540C	SW-846 8270C
Carbazole	2	SW-846 3540C	SW-846 8270C
Chrysene	2	SW-846 3540C	SW-846 8270C
Dibenz(a,h)anthracene	2	SW-846 3540C	SW-846 8270C
Dibenzofuran	2	SW-846 3540C	SW-846 8270C
Diethylphthalate	2	SW-846 3540C	SW-846 8270C
Dimethylphthalate	2	SW-846 3540C	SW-846 8270C

Table 10. (continued).

Analysis	Recommended Detection Limit	Preparation Method	Analysis Method
TCLP Extraction	Not Applicable	SW-846 1311	Not Applicable
Di-n-butylphthalate	2	SW-846 3540C	SW-846 8270C
Di-n-octylphthalate	2	SW-846 3540C	SW-846 8270C
Fluoranthene	2	SW-846 3540C	SW-846 8270C
Fluorene	2	SW-846 3540C	SW-846 8270C
Hexachlorobenzene	2	SW-846 3540C	SW-846 8270C
Hexachlorobutadiene	2	SW-846 3540C	SW-846 8270C
Hexachlorocyclopentadiene	2	SW-846 3540C	SW-846 8270C
Hexachloroethane	2	SW-846 3540C	SW-846 8270C
Indeno(1,2,3-cd)pyrene	2	SW-846 3540C	SW-846 8270C
Isophorone	2	SW-846 3540C	SW-846 8270C
Naphthalene	2	SW-846 3540C	SW-846 8270C
Nitrobenzene	2	SW-846 3540C	SW-846 8270C
N-Nitrosodimethylamine	2	SW-846 3540C	SW-846 8270C
N-Nitroso-di-n-propylamine	2	SW-846 3540C	SW-846 8270C
N-Nitrosodiphenylamine	2	SW-846 3540C	SW-846 8270C
Pentachlorophenol	5	SW-846 3540C	SW-846 8270C
Phenanthrene	2	SW-846 3540C	SW-846 8270C
Phenol	2	SW-846 3540C	SW-846 8270C
Pyrene	2	SW-846 3540C	SW-846 8270C
Pyridine	4	SW-846 3540C	SW-846 8270C
Tri-n-butylphosphate	5	SW-846 3540C	SW-846 8270C
Aroclors (PCBs)	(mg/kg)		
Aroclor-1016	0.4	SW-846 3540C	SW-846 8082
Aroclor-1221	0.2	SW-846 3540C	SW-846 8082
Aroclor-1232	0.2	SW-846 3540C	SW-846 8082
Aroclor-1242	0.2	SW-846 3540C	SW-846 8082
Aroclor-1248	0.2	SW-846 3540C	SW-846 8082
Aroclor-1254	0.2	SW-846 3540C	SW-846 8082
Aroclor-1260	0.2	SW-846 3540C	SW-846 8082

a. Based on ^{137}Cs ; all other gamma isotopes will have a detection limit commensurate with their photon yield and energy as related to the ^{137}Cs detection limit.

b. The estimated quantitation limit (EQL) is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5–10 times the method detection limit. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes, the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided as an example from SW-846 (EPA 1998) and may not always be achievable.

Table 11. Sample preparation, analytical methods, and recommended detection limits—liquids.

Analysis	Recommended Detection Limit	Preparation Method	Analysis Method
Anions	(mg/L)		
Chloride	0.2	SW-846 9056	SW-846 9056
Fluoride	0.05	SW-846 9056	SW-846 9056
Nitrate	0.02	SW-846 9056	SW-846 9056
Phosphate	0.03	SW-846 9056	SW-846 9056
Sulfate	0.2	SW-846 9056	SW-846 9056
Total Metals	(mg/L)^a		
Aluminum	0.3	SW-846 3010A	SW-846 6010B
Antimony	0.2	SW-846 3010A	SW-846 6010B
Arsenic	0.01	SW-846 3010A	SW-846 6010B
Barium	0.01	SW-846 3010A	SW-846 6010B
Beryllium	0.002	SW-846 3010A	SW-846 6010B
Cadmium	0.02	SW-846 3010A	SW-846 6010B
Calcium	0.07	SW-846 3010A	SW-846 6010B
Chromium	0.05	SW-846 3010A	SW-846 6010B
Cobalt	0.05	SW-846 3010A	SW-846 6010B
Copper	0.04	SW-846 3010A	SW-846 6010B
Iron	0.04	SW-846 3010A	SW-846 6010B
Lead	0.3	SW-846 3010A	SW-846 6010B
Manganese	0.01	SW-846 3010A	SW-846 6010B
Mercury	0.002	SW 846 7470A	SW-846 7470A
Nickel	0.1	SW-846 3010A	SW-846 6010B
Selenium	0.02	SW-846 3010A	SW-846 6010B
Silver	0.05	SW-846 3010A	SW-846 6010B
Thallium	0.3	SW-846 3010A	SW-846 6010B
Vanadium	0.05	SW-846 3010A	SW-846 6010B
Zinc	0.01	SW-846 3010A	SW-846 6010B
Radionuclides	(pCi/L)		
²⁴¹ Am	0.2	ER-SOW-394	ER-SOW-394
¹⁴ C	3	ER-SOW-394	ER-SOW-394
²⁴⁴ Cm	0.2	ER-SOW-394	ER-SOW-394
³ H	400	ER-SOW-394	ER-SOW-394
¹²⁹ I	1	ER-SOW-394	ER-SOW-394
⁶³ Ni	5	ER-SOW-394	ER-SOW-394
²³⁷ Np	0.2	ER-SOW-394	ER-SOW-394
^{238, 239/240} Pu	0.2	ER-SOW-394	ER-SOW-394
²⁴¹ Pu	10	ER-SOW-394	ER-SOW-394
⁹⁰ Sr	1	ER-SOW-394	ER-SOW-394
⁹⁹ Tc	10 ^b	ER-SOW-394	ER-SOW-394
U isotopic	0.5	ER-SOW-394	ER-SOW-394

Table 11. (continued).

Analysis	Recommended Detection Limit	Preparation Method	Analysis Method
Gamma-emitting Radionuclides: ⁶⁰ Co, ¹³⁴ , ¹³⁷ Cs, ⁹⁴ Nb, ¹⁵⁴ , ¹⁵⁵ Eu	30 ^c	ER-SOW-394	ER-SOW-394
Organic Constituents			
Volatiles^d	(mg/L)		
2-Butanone	0.01	SW-846 5030B	SW-846 8260B
1,2-Dichlorobenzene	0.01	SW-846 5030B	SW-846 8260B
1,3-Dichlorobenzene	0.01	SW-846 5030B	SW-846 8260B
1,4-Dichlorobenzene	0.01	SW-846 5030B	SW-846 8260B
1,1-Dichloroethene	0.01	SW-846 5030B	SW-846 8260B
1,2-Dibromoethane	0.01	SW-846 5030B	SW-846 8260B
1,2-Dibromo-3-chloropropane	0.01	SW-846 5030B	SW-846 8260B
1,2-Dichloropropane	0.01	SW-846 5030B	SW-846 8260B
2-Hexanone	0.01	SW-846 5030B	SW-846 8260B
4-Methyl-2-pentanone	0.01	SW-846 5030B	SW-846 8260B
1,1,2,2-Tetrachloroethane	0.01	SW-846 5030B	SW-846 8260B
1,2,4-Trichlorobenzene	0.01	SW-846 5030B	SW-846 8260B
1,1,1-Trichloroethane	0.01	SW-846 5030B	SW-846 8260B
1,1,2-Trichloroethane	0.01	SW-846 5030B	SW-846 8260B
1,1,2-Trichloro-1,2,2-Trifluoroethane	0.01	SW-846 5030B	SW-846 8260B
Acetone	0.01	SW-846 5030B	SW-846 8260B
Benzene	0.01	SW-846 5030B	SW-846 8260B
Bromodichloromethane	0.01	SW-846 5030B	SW-846 8260B
Bromoform	0.01	SW-846 5030B	SW-846 8260B
Bromomethane	0.01	SW-846 5030B	SW-846 8260B
Carbon Disulfide	0.01	SW-846 5030B	SW-846 8260B
Carbon Tetrachloride	0.01	SW-846 5030B	SW-846 8260B
Chlorobenzene	0.01	SW-846 5030B	SW-846 8260B
Chloroethane	0.01	SW-846 5030B	SW-846 8260B
Chloroform	0.01	SW-846 5030B	SW-846 8260B
Chloromethane	0.01	SW-846 5030B	SW-846 8260B
cis-1,2-Dichloroethene	0.01	SW-846 5030B	SW-846 8260B
cis-1,3-Dichloropropene	0.01	SW-846 5030B	SW-846 8260B
Cyclohexane	0.01	SW-846 5030B	SW-846 8260B
Cyclohexanone	0.007	SW-846 5030B	SW-846 8260B
Dibromochloromethane	0.01	SW-846 5030B	SW-846 8260B
Dichlorodifluoromethane	0.01	SW-846 5030B	SW-846 8260B
Ethyl Acetate	0.025	SW-846 5030B	SW-846 8260B
Ethylbenzene	0.01	SW-846 5030B	SW-846 8260B
Isopropylbenzene	0.01	SW-846 5030B	SW-846 8260B
Methanol	0.025	SW-846 8015B	SW-846 8015B
Methyl Acetate	0.01	SW-846 5030B	SW-846 8260B

Table 11. (continued).

Analysis	Recommended Detection Limit	Preparation Method	Analysis Method
Methylcyclohexane	0.01	SW-846 5030B	SW-846 8260B
Methylene Chloride	0.01	SW-846 5030B	SW-846 8260B
Styrene	0.01	SW-846 5030B	SW-846 8260B
Tetrachloroethene	0.01	SW-846 5030B	SW-846 8260B
Toluene	0.01	SW-846 5030B	SW-846 8260B
trans-1,2-Dichloroethene	0.01	SW-846 5030B	SW-846 8260B
trans-1,3-Dichloropropene	0.01	SW-846 5030B	SW-846 8260B
Trichloroethene	0.01	SW-846 5030B	SW-846 8260B
Trichlorofluoromethane	0.01	SW-846 5030B	SW-846 8260B
Vinyl Chloride	0.01	SW-846 5030B	SW-846 8260B
Xylenes (Total)	0.01	SW-846 5030B	SW-846 8260B
Semivolatiles	(mg/L)		
1,1'-Biphenyl	0.01	SW-846 3520C	SW-846 8270C
4-Bromophenyl-phenylether	0.01	SW-846 3520C	SW-846 8270C
4-Chloroaniline	0.01	SW-846 3520C	SW-846 8270C
4-Chlorophenyl-phenylether	0.01	SW-846 3520C	SW-846 8270C
4-Chloro-3-methylphenol	0.01	SW-846 3520C	SW-846 8270C
3,3'-Dichlorobenzidine	0.01	SW-846 3520C	SW-846 8270C
2,4-Dichlorophenol	0.01	SW-846 3520C	SW-846 8270C
2,4-Dimethylphenol	0.01	SW-846 3520C	SW-846 8270C
2,4-Dinitrophenol	0.025	SW-846 3520C	SW-846 8270C
2,4-Dinitrotoluene	0.01	SW-846 3520C	SW-846 8270C
2,6-Dinitrotoluene	0.01	SW-846 3520C	SW-846 8270C
4,6-Dinitro-2-methylphenol	0.025	SW-846 3520C	SW-846 8270C
2-Chloronaphthalene	0.01	SW-846 3520C	SW-846 8270C
2-Chlorophenol	0.01	SW-846 3520C	SW-846 8270C
2-Methylnaphthalene	0.01	SW-846 3520C	SW-846 8270C
2-Methylphenol	0.01	SW-846 3520C	SW-846 8270C
4-Methylphenol	0.01	SW-846 3520C	SW-846 8270C
2-Nitroaniline	0.025	SW-846 3520C	SW-846 8270C
3-Nitroaniline	0.025	SW-846 3520C	SW-846 8270C
4-Nitroaniline	0.025	SW-846 3520C	SW-846 8270C
2-Nitrophenol	0.01	SW-846 3520C	SW-846 8270C
4-Nitrophenol	0.025	SW-846 3520C	SW-846 8270C
2,2'-Oxybis (1-Chloropropane)	0.01	SW-846 3520C	SW-846 8270C
2,4,5-Trichlorophenol	0.025	SW-846 3520C	SW-846 8270C
2,4,6-Trichlorophenol	0.01	SW-846 3520C	SW-846 8270C
Acenaphthene	0.01	SW-846 3520C	SW-846 8270C
Acenaphthylene	0.01	SW-846 3520C	SW-846 8270C
Acetophenone	0.01	SW-846 3520C	SW-846 8270C
Anthracene	0.01	SW-846 3520C	SW-846 8270C

Table 11. (continued).

Analysis	Recommended Detection Limit	Preparation Method	Analysis Method
Atrazine	0.01	SW-846 3520C	SW-846 8270C
Benzaldehyde	0.01	SW-846 3520C	SW-846 8270C
Benzo(a)anthracene	0.01	SW-846 3520C	SW-846 8270C
Benzo(a)pyrene	0.01	SW-846 3520C	SW-846 8270C
Benzo(b)fluoranthene	0.01	SW-846 3520C	SW-846 8270C
Benzo(g,h,i)perylene	0.01	SW-846 3520C	SW-846 8270C
Benzo(k)fluoranthene	0.01	SW-846 3520C	SW-846 8270C
bis-(2-Chloroethoxy) methane	0.01	SW-846 3520C	SW-846 8270C
bis-(2-Chloroethyl)ether	0.01	SW-846 3520C	SW-846 8270C
bis-(2-Ethylhexyl)phthalate	0.01	SW-846 3520C	SW-846 8270C
Butylbenzylphthalate	0.01	SW-846 3520C	SW-846 8270C
Caprolactam	0.01	SW-846 3520C	SW-846 8270C
Carbazole	0.01	SW-846 3520C	SW-846 8270C
Chrysene	0.01	SW-846 3520C	SW-846 8270C
Dibenz(a,h)anthracene	0.01	SW-846 3520C	SW-846 8270C
Dibenzofuran	0.01	SW-846 3520C	SW-846 8270C
Diethylphthalate	0.01	SW-846 3520C	SW-846 8270C
Dimethylphthalate	0.01	SW-846 3520C	SW-846 8270C
Di-n-butylphthalate	0.01	SW-846 3520C	SW-846 8270C
Di-n-octylphthalate	0.01	SW-846 3520C	SW-846 8270C
Fluoranthene	0.01	SW-846 3520C	SW-846 8270C
Fluorene	0.01	SW-846 3520C	SW-846 8270C
Hexachlorobenzene	0.01	SW-846 3520C	SW-846 8270C
Hexachlorobutadiene	0.01	SW-846 3520C	SW-846 8270C
Hexachlorocyclopentadiene	0.01	SW-846 3520C	SW-846 8270C
Hexachloroethane	0.01	SW-846 3520C	SW-846 8270C
Indeno(1,2,3-cd)pyrene	0.01	SW-846 3520C	SW-846 8270C
Isophorone	0.01	SW-846 3520C	SW-846 8270C
Naphthalene	0.01	SW-846 3520C	SW-846 8270C
Nitrobenzene	0.01	SW-846 3520C	SW-846 8270C
N-Nitrosodimethylamine	0.01	SW-846 3520C	SW-846 8270C
N-Nitroso-di-n-propylamine	0.01	SW-846 3520C	SW-846 8270C
N-Nitrosodiphenylamine	0.01	SW-846 3520C	SW-846 8270C
Pentachlorophenol	0.025	SW-846 3520C	SW-846 8270C
Phenanthrene	0.01	SW-846 3520C	SW-846 8270C
Phenol	0.01	SW-846 3520C	SW-846 8270C
Pyrene	0.01	SW-846 3520C	SW-846 8270C
Pyridine	0.02	SW-864 3520C	SW-846 8015B
Tri-n-butylphosphate	0.025	SW-846 3520C	SW-846 8270C

Table 11. (continued).

Analysis	Recommended Detection Limit	Preparation Method	Analysis Method
Aroclors (PCBs) Water	(mg/L)		
Aroclor-1016	0.001	SW-846 3520C	SW-846-8082
Aroclor-1221	0.002	SW-846 3520C	SW-846-8082
Aroclor-1232	0.001	SW-846 3520C	SW-846-8082
Aroclor-1242	0.001	SW-846 3520C	SW-846-8082
Aroclor-1248	0.001	SW-846 3520C	SW-846-8082
Aroclor-1254	0.001	SW-846 3520C	SW-846-8082
Aroclor-1260	0.001	SW-846 3520C	SW-846-8082

a. The method detection limits for metals analyses conducted on the aqueous post-decontamination residuals is estimated by multiplying published instrument detection limits by ten.

b. May require analysis by inductively coupled plasma/mass spectrometry to achieve the detection limit because of interference introduced by the activity of other radioactive isotopes present.

c. Based on ^{137}Cs ; all other gamma isotopes shall have a detection limit commensurate with their photon yield and energy as related to the ^{137}Cs detection limit.

d. The estimated quantitation limit (EQL) is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5–10 times the method detection limit. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes, the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided as an example from SW-846 (EPA 1998) and may not always be achievable.

8. INSTRUMENT CALIBRATION PROCEDURES

To ensure that sampling and analysis activities obtain the most accurate and precise information possible, field equipment and laboratory instrumentation must be calibrated according to both manufacturer specifications and the appropriate analytical method specifications.

8.1 Laboratory Instrument Calibration

Laboratory instrumentation will be calibrated in accordance with each of the specified analytical methods (Table 9). The laboratory QA plan shall include requirements for calibrations when specifications are not listed in analytical methods. Calibrations that are typically not called out in analytical methods include ancillary laboratory equipment (e.g., analytical balances, pipettes, and pH meters) and verification of reference standards used for calibration and standard preparation. Laboratory documentation will include calibration techniques and sequential calibration actions, performance tolerances provided by the specific analytical method, and calibration dates and frequency. In addition, records for all laboratory-prepared standards will be maintained and provided with each data deliverable. Instrument responses for gas chromatography (GC)/mass spectrometry (MS), GC retention time window definitions, and documentation of calibration check precision for GC and GC/MS systems will be reported in each deliverable. Standard reference materials used to perform calibration checks associated with both inorganic target analytes and radiochemical parameters will be prepared using an independent source for the standard materials from that used to prepare the calibration standards. The results of these calibration checks will be reported with each data deliverable.

All analytical methods prescribed in Table 9 have specifications for equipment checks and instrument calibrations. The laboratory will comply with all method-specific calibration requirements for all requested parameters. If an instrument calibration or equipment fails, the instrument will be recalibrated and all affected samples will be analyzed using an acceptable calibration.

8.2 Field Equipment Calibration/Setup

The FTL will work closely with the operations personnel in charge of the submersible pump to ensure that it is operating as recommended by the manufacturer and/or according to the design specifications. The required pre-sampling inspections will evaluate the submersible pump mechanisms to ensure that they are functioning properly before the submersible pump is placed into the tanks. Corrective actions for the repair or maintenance of the submersible pump will be immediate and will be confirmed by the PM before sample collection. All field calibrations will be documented in a field instrument calibration/standardization logbook.

8.3 Preventative Maintenance Procedures and Frequency

Field equipment will be managed using a calibration program compliant with all INEEL procedures. All laboratory equipment will be maintained to a level such that each piece of equipment and each laboratory instrument can meet method-specific QA/QC tolerances. Maintenance will be performed under the supervision of qualified personnel on all laboratory instrumentation in accordance with the manufacturer's specifications, laboratory QA plan, and standard operating procedures.

Preventive maintenance of field equipment will be conducted in accordance with appropriate facility standard operating procedures. *EPA Requirements for Quality Assurance Project Plans* (EPA 2001) and ER-SOW-394 (2002) require that all activities not governed by specific analytical procedures be completed under approved standard operating procedures. If standard operating procedures

governing the inspection and maintenance of sampling equipment do not presently exist, they will be developed to ensure that sampling activities are conducted using equipment that is performing within manufacturer or design specifications.

Equipment used by INTEC ESH&Q oversight personnel will be evaluated, maintained, and operated within the manufacturers' specifications for each type of field or monitoring equipment.

9. DATA VALIDATION AND REPORTING

The collection of data in the field and by the laboratory is the first of several steps in evaluating conditions at a project site. After the data are collected, a series of evaluations and data reduction steps must be conducted to ensure that the data are acceptable and that the information is in a form that is practical for the end users.

9.1 Data Reduction

Data reduction is the process of converting raw data or instrument data into a usable form for evaluation by project personnel. Reduction of environmental data will take place at the laboratory. The data reduction activities performed at the laboratory convert the data into a form more usable for interpretive purposes for environmental risk assessment and verification of closure design.

Laboratory data reduction involves converting the outputs of the analytical instruments into sample and QC results. Laboratory reduction will be performed as defined in the analytical method. Laboratory deliverables include raw data and reduced data. This form of laboratory reporting will: (a) ensure complete documentation of all aspects of laboratory analysis, (b) permit independent verification of reported results, (c) provide a form of data that is technically and legally defensible, and (d) ensure that end data users can be completely confident in the results they deem usable.

Further data reduction may be necessary for use at the project level. When this is necessary, the PM will determine the final data uses and parameter needs and will provide data sets in the form that project personnel require to complete their tasks. Examples of additional data reduction tasks include unit conversions and use of the data to perform sum-of-the-fractions calculations defined in 10 CFR 61.55(a)(7) (2003).

Scientists and regulators within EPA, DOE Headquarters, DOE Idaho Operations Office, and IDEQ may review the data to ensure compliance with HWMA/RCRA and DOE closure requirements. Individual regulators will submit their requests to the PM for any data sets required to evaluate the post-decontamination characterization effort. The PM will provide requested information to regulators in the most usable form possible.

9.2 Data Validation

Analytical data validation is the comparison of analytical results versus the requirements established by the analytical method. Validation involves evaluation of all sample-specific information generated from sample collection to receipt of the final data package by the PM. Data validation is used to determine whether the analytical data are technically and legally defensible and reliable. The applicable analytical method QC guidelines will be used to validate the data with the exception of radioanalytical data, which will be validated exclusively using TPR-80, "Radioanalytical Data Validation" (1997). Data validation is one step of the DQA process that is used to determine whether the data meet the DQOs of the project. Additional steps of the DQA process are discussed in Section 9.3.

The final product of the validation process is the validation report. The validation report communicates the quality and usability of the data to the decision-makers. The validation report will contain an itemized discussion of the validation process and results. Copies of the data forms annotated for qualification as discussed in the validation report will be attached to the report.

9.3 Data Quality Assessment

The DQA process is used to determine whether the data meet the project DQOs. Following data validation, the DQA process involves data plotting, testing for outlying data points, and statistical hypothesis testing relative to the null and alternative hypotheses stated in the DQOs. The outcome of the DQA process is a DQA report documenting that the statistical hypothesis testing suggests that the null hypothesis is accurate, that the null hypothesis has been rejected, or that not enough data exist to make a determinative conclusion based on the hypothesis test used. In this latter case, either additional data must be collected to support the statistical hypothesis testing or the data user must make a decision with higher uncertainty than the levels expressed in the DQOs.

As stated in the discussion of completeness, data that are not necessarily invalid may be flagged during the data validation process. Flagged data are reviewed during the DQA process to determine whether the validation flags affect the intended use of the data. The determination of whether or not flagged data are used in statistical hypothesis testing is documented in the DQA report.

9.4 Data Use

Following data validation and DQA, the statistics generated during DQA will be used to make decisions relative to HWMA/RCRA clean closure and DOE Tier 1 closure. The data generated will be used to determine the concentration variance and sample mean (\bar{x}) for each constituent of concern. For hazardous constituents, the data also will be used to calculate the 95% UCL of the sample mean, and that value will be used as a conservative estimate of the population mean (μ). The concentration corresponding to the 95% UCL will be compared to the action levels in the HWMA/RCRA closure plan (DOE-ID 2004) to determine if the clean-closure performance standards have been met within the decision errors specified in the DQOs. For radionuclide analyses, the sample mean (\bar{x}) as represented by the 95% UCL will be used to verify the PA (DOE-ID 2003c).

9.5 Reporting

The laboratory may use its standard report forms when assembling the final standard plus raw data deliverable data package documentation. However, each deliverable must conform to the criteria specified in ER-SOW-394 (2002) and the applicable laboratory contract.

The standard plus raw data deliverables include all pertinent raw data, extraction notes, standard preparation, instrument printouts, and standard reference material certificates, in accordance with ER-SOW-394. This SOW, prepared by the INEEL Sample and Analysis Management Office, has become the standard means by which analytical data deliverable requirements are defined by INEEL projects to both INEEL laboratories and commercial laboratories used by the INEEL.

10. INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

To adequately assess the quality of sampling techniques and the cleanliness of sampling and shipping methods, and to help assess laboratory accuracy and precision, field QA/QC samples will be submitted with natural samples at the time of custody transfer to the laboratory. Sampling conditions during the WM-103 through WM-106 and WM-181 post-decontamination tank heel characterization may be unconventional, and field QC will be difficult to incorporate into the sampling process. However, depending on conditions, some field QC can be applied and will be collected. For this reason, it will be critical for laboratory QA/QC procedures and tolerances to be closely followed and met whenever possible. The following sections outline specific QC checks that will take place for this project.

10.1 Laboratory Quality Control

Strict adherence to laboratory QA/QC procedures and analytical method tolerances are critical to obtaining high-quality laboratory data. Each analysis conducted under the WM-103 through WM-106 and WM-181 post-decontamination characterization will strictly adhere to all QA/QC procedures, QA/QC control limits, and method-specific corrective actions.

NOTE: Because of the negative pressure in which samples are collected (the vacuum used for sample collection) and elevated temperatures in the tanks and the RAL hot cell, all VOA and SVOA results have the potential for low bias.

10.2 Field Quality Control

Field QC usually is accomplished by using approved sampling procedures and is monitored by using trip and field blanks as described in Section 5.1.6, "Sample Transport." However, as stated in Section 5.1.6, field blanks will not be collected; trip blanks will be collected if aliquots of the samples will be shipped off-site for VOC analyses.

10.3 Inspection/Acceptance Requirements for Supplies and Consumables

Disposable sampling equipment will be checked before use to ensure it is made of material appropriate for the media being sampled. Sample containers will be obtained from vendors that certify the cleaning protocol used is appropriate for the analyses to be performed on the sample. Reagents used for sample preservation will be checked to ensure they are of the appropriate grade before use. Inspection and acceptance of these items will be documented in field logbooks or, when certifications are provided by the manufacturer, maintained in project files to ensure availability of these records.

11. SYSTEM AND PERFORMANCE ASSESSMENTS, FREQUENCY AND CORRECTIVE ACTIONS

It is not a requirement of this SAP that a formal audit of the analytical laboratory be performed before commencing with the WM-103 through WM-106 and WM-181 tank heels post-decontamination characterization. However, if deviations from the procedures outlined in this SAP are suspected during analysis, the PM and the PQAO should review the laboratory procedures that were used to obtain project data. In addition, an onsite meeting at the laboratory is encouraged to examine all procedures in action, to examine the facilities that will be used to complete data-gathering activities, and to discuss the technical project activities and intended data uses with laboratory personnel.

11.1 System and Performance Assessments

A system assessment is an evaluation of an entire system to ensure it will meet the requirements of the project. An example of a system assessment is an onsite laboratory audit that ensures the sample receiving, sample storage, sample analysis, data reduction, and documentation procedures used at the laboratory will meet the requirements of the project. A PA is the evaluation of the performance of one aspect of a system. An example of a PA is the insertion of performance evaluation samples to test the laboratory system. Performance evaluation samples are samples containing analytes of interest at known concentrations.

11.2 Corrective Action

Corrective action procedures are implemented whenever sampling, field monitoring, or laboratory analysis results do not meet the required QA/QC standards. The types of corrective action applicable to environmental analysis are laboratory corrective action(s) and field corrective action(s).

11.2.1 Laboratory Corrective Action

The laboratory manager, laboratory QA officer, laboratory analysts, PM, and PQAO will be responsible for ensuring that all laboratory QA/QC procedures are followed. Situations requiring corrective action and the type of correction required will be stated in the analytical method or the laboratory SOW. The laboratory will use internal QA plans and standard operating procedures to complete all corrective actions identified both internally and externally. Completion of corrective actions will require notification to the PM or the PQAO of any laboratory situation that may affect the usability of the data. If notified of a laboratory nonconformance for which the laboratory seeks the project's required corrective action, the PM or PQAO will

- Notify the PQAO/PM of the situation
- Devise a reasonable corrective action in conjunction with the laboratory staff and the PQAO/PM
- Formally request the laboratory to implement the corrective action.

The PQAO and the laboratory QA officer will be responsible for monitoring the effectiveness of all corrective actions. The PQAO will report directly to the PM and INEEL management regarding problems or deviations observed, corrective actions proposed, and the effectiveness of ongoing corrective actions.

11.2.2 Field Corrective Action

The FTL and PM are responsible for ensuring all field procedures are followed completely and that field personnel are trained adequately. The FTL and PM must document situations that may impair the usability of the samples and/or data in the field logbook. The FTL will note any deviations that occur from the standard procedures for sample collection, COC, sample transport, or monitoring. The FTL will also be responsible for coordinating all activities related to the use of field monitoring equipment, such as dosimeters and industrial hygiene equipment. Oversight personnel from INTEC ESH&Q will provide any notations to the logbook to document noncompliant measurements taken during field sampling. Ultimately, the PM or FTL (at the discretion of the PM) will be responsible for communicating field corrective action procedures, documenting all deviations from procedure, and ensuring that immediate corrective actions are applied to field activities.

11.3 Reports to Management

The FTL and PM are responsible for ensuring all field procedures are completely followed and that field personnel are adequately trained. The FTL and PM must document situations that may impair the usability of the samples and/or data in the field logbook. The FTL will note any deviations that occur from the standard procedures for sample collection, COC, sample transport, or monitoring. The FTL will communicate any deviations to the Environmental Affairs closure PM, who will discuss these deviations with the independent PE to ensure any deviations are minor and do not affect implementation of the approved closure plan. The FTL will also be responsible for coordinating all activities related to the use of field monitoring equipment (e.g., dosimeters and industrial hygiene equipment). The RCT and IH will provide any notations to document out-of-compliance measurements taken during field sampling. Ultimately, the PM or FTL (at the discretion of the PM) will be responsible for effectively communicating field corrective action procedures, documenting all deviations from procedures, and ensuring that immediate corrective actions are applied to field activities.

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Appendix A

Crosswalk between the Environmental Protection Agency QAPP and FSP Requirements and the SAP for Post-Decontamination Characterization of WM-103 through WM-106 and WM-181 Tank Residuals

Appendix A

Crosswalk between the Environmental Protection Agency QAPP and FSP Requirements and the SAP for Post-Decontamination Characterization of WM-103 through WM-106 and WM-181 Tank Residuals

In 1989, EPA published *Guidance for Conducting Remedial Investigations and Feasibility Studies under the CERCLA, Interim Final* (EPA 1989). This document stated that a SAP consisted of two separate documents, an FSP and a QAPP. In 1998 (revised in 2002), EPA published *Guidance for Quality Assurance Project Plans* (EPA 2002), and in 2001, EPA published *EPA Requirements for Quality Assurance Project Plans* (EPA 2001). These recent documents expand on the guidance provided in the 1989 EPA guidance. Most notably, the 2001 and 2002 documents take the elements defined in the 1989 EPA guidance, which previously required both an FSP and a QAPP to implement, and combine them into one document. Thus, EPA's 2001 and 2002 direction implies that only a single QAPP document is required for each sampling and analysis activity. To alleviate confusion between the old and new nomenclature, and to aid readers in locating specific information of interest, two crosswalk tables are given here, comparing all three EPA documents and the SAP for Tanks WM-103 through WM-106 and WM-181. Table A-1 compares the QAPP elements and Table A-2 compares the FSP elements.

Table A-1. Comparison of QAPP elements in EPA QA/R-5 requirements and QA/G-5 guidance documents to Conducting Remedial Investigations and Feasibility Studies under CERCLA and the elements in the sampling and analysis plan for WM-103 through WM-106 and WM-181 tank residuals.

<i>EPA QA/R-5 Requirements/ EPA QA/G-5 Guidance QAPP Elements</i>		<i>Conducting Remedial Investigations and Feasibility Studies under CERCLA QAPP Elements^a</i>		<i>Applicable Sections in the SAP for WM-103 through WM-106 and WM-181 Tank Residuals^a</i>	
A. Project Management					
A1.	Title and Approval Sheet		Title Page		Title and Approval Sheet
A2.	Table of Contents		Table of Contents		Table of Contents in INEEL Document Control Format
A3.	Distribution List		N/A		N/A
A4.	Project/Task Organization	2.	Project Organization and Responsibilities	2.	Project Organization and Responsibilities
A5.	Problem Definition/Background	1.	Project Description	1. 3.1.1	Project Description Problem Statement
A6.	Project Task Description/Schedule	1.	Project Description	1. 3.1.1 3.1.4	Project Description Problem Statement Study Boundaries
A7.	Quality Objectives and Criteria	3.	QA Objectives for Measurement	3.	Quality Objectives and Criteria for Measurement Data
A8.	Special Training Requirements/Certification		N/A		N/A
A9.	Documentation and Records		N/A	4.	Documentation and Data Management
B. Measurement/Data Acquisition					
B1.	Sampling Process Design (Experimental Design)		N/A	3.1 5.	Data Quality Objectives Sampling Process Design
B2.	Sampling Methods	4.	Sampling Procedures	6.	Sampling Procedures
B3.	Sample Handling and Custody	5.	Sample Custody	4.1.1 5.1.5 5.1.6	Field Operations Records Sample Containers Sample Transport
B4.	Analytical Methods	7.	Analytical Procedures	7. 8.1	Analytical Methods Laboratory Instrument Calibration
B5.	Quality Control	9.	Internal Quality Control	10.	Internal Quality Control Checks and Frequency
B6.	Instrument/Equipment Testing, Inspection, and Maintenance	6. 11.	Calibration Procedures Preventive Maintenance	8. 10.1	Instrument Calibration Procedures Laboratory Quality Control

Table A-1. (continued).

<i>EPA QA/R-5 Requirements/ EPA QA/G-5 Guidance QAPP Elements</i>		<i>Conducting Remedial Investigations and Feasibility Studies under CERCLA QAPP Elements^a</i>		<i>Applicable Sections in the SAP for WM-103 through WM-106 and WM-181 Tank Residuals^a</i>	
B7.	Instrument/Equipment Calibration and Frequency	7.	Analytical Procedures	8.	Instrument Calibration Procedures
		9.	Internal Quality Control		
B8.	Inspection/Acceptance of Supplies and Consumables	9.	Internal Quality Control	10.3	Inspection/Acceptance Requirements for Supplies and Consumables
B9.	Data Acquisition Requirements (Non-Direct Measurements)	12.	Data Assessment Procedures	3.1.3	Decision Inputs
B10.	Data Management	8.	Data Reduction, Validation, and Reporting	4.	Documentation and Data Management
C. Assessment/Oversight					
C1.	Assessments and Response Actions	10.	Performance and System Audits	11.	System and Performance Assessments, Frequency, and Corrective Actions
		13.	Corrective Actions		
C2.	Reports to Management	14.	Quality Assurance Reports	11.3	Reports to Management
D. Data Validation and Usability					
D1.	Data Review, Verification, and Validation	8.	Data Reduction, Validation, and Reporting	9.	Data Validation and Reporting
		12.	Data Assessment Procedures		
D2.	Verification and Validation Methods	12.	Data Assessment Procedures	9.	Data Validation and Reporting
D3.	Reconciliation with Data Quality Objectives/User Requirements	12.	Data Assessment Procedures	9.3	Data Quality Assessment

a. N/A = Not available.

Table A-2. Comparison of FSP elements in EPA QA/R-5 requirements and QA/G-5 guidance documents to Conducting Remedial Investigations and Feasibility Studies under CERCLA and the elements in the sampling and analysis plan for WM-103 through WM-106 and WM-181 tank residuals.

<i>Conducting Remedial Investigations and Feasibility Studies under CERCLA</i> FSP Elements		EPA QA/R-5 Requirements/ EPA QA/G-5 Guidance QAPP Elements		Applicable Sections in the Sampling and Analysis Plan for WM-103 through WM-106 and WM-181 Tank Residuals	
1.	Site Background	A5.	Problem Definition/Background	1.	Project Description
		A6.	Project Task Description/Schedule	1.2	Background
2.	Sampling Objectives	A5.	Problem Definition/Background	1.	Project Description
		A6.	Project Task Description/Schedule	3.1.1	Problem Statement
				3.1.2	Decision Statement
				3.1.3	Decision Inputs
3.	Sample Location and Frequency			3.1.4	Study Boundaries
		B1.	Sampling Process Design (Experimental Design)	3.1.7	Design Optimization
4.	Sample Designation			5.1.2	Sample Location and Frequency
		A9.	Documentation and Records	4.1.1	Field Operations Records
5.	Sampling Equipment and Procedures	B3.	Sample Handling and Custody		
		B1.	Sampling Process Design (Experimental Design)	5.	Sampling Process Design
		B2.	Sampling Methods	6.	Sampling Procedures
		B6.	Instrument/Equipment Testing, Inspection, and Maintenance		
6.	Sample Handling and Analysis	B3.	Sample Handling and Custody	5.1.5	Sample Containers
		B4.	Analytical Methods	5.1.6	Sample Transport
				7.	Analytical Methods
				8.	Instrument Calibration Procedures

REFERENCES

- EPA, 1998, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Revision 5, Office of Solid Waste, Washington D.C., April 1998.
- EPA, 2001, *EPA Requirements for Quality Assurance Project Plans*, EPA QA/R-5, EPA/240/B-01/003, Office of Environmental Information, Washington D.C., March 2001.
- EPA, 2002, *Guidance for Quality Assurance Project Plans*, EPA QA/G-5, EPA/240/R-02/009, Office of Environmental Information, Washington, D.C., December 2002.